







TERRESTRIAL AND ECOLOGICAL RISK ASSESSMENT AT U.S. ARMY ABERDEEN PROVING GROUND QUALITY ASSURANCE PROJECT PLAN, VOLUME I

FINAL DOCUMENT

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QUALITY ASSURANCE PROJECT PLAN FOR TASK ORDER NO. 10 TERRESTRIAL AND ECOLOGICAL RISK ASSESSMENT AT U.S. ARMY ABERDEEN PROVING GROUND

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SECTION 1.0

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1.0 INTRODUCTION

The United States Army Environmental Center (USAEC), formerly the United States Army Toxic and Hazardous Materials Agency (USATHAMA), has tasked ICF Kaiser Engineers, Inc. (ICF KE) to collect data to conduct a Terrestrial and Ecological Risk Assessment (TERA) at U.S. Army Aberdeen Proving Ground (APG) in accordance with guidance from the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA). This work will be performed under Contract No. DAAA15-91-D-0014, Task Order No. 10.

The Quality Assurance Project Plan (QAPjP) delineates the purpose, policies, standard operating procedures (SOPs) and organization of the QA program which will be used to establish the integrity of APG TERA project activities. This QAPjP was written to complement the Risk and Biological Impact Assessment (ICF KE 1992) where workplans for eighteen study areas at APG were developed. The work proposed in the workplans will be performed under Task No. 10. The procedures detailed in this QAPjP are in compliance with the Environmental Protection Agency (USEPA) Region III and with the USATHAMA Installation Restoration Quality Assurance Program (1990), where applicable.

The QAPjP is divided into thirteen sections. Project description, organization, and responsibilities are delineated in Sections 2 and 3. Responsibilities for field and laboratory activities are provided as well as a list of key individuals. Section 4 defines the data quality objectives for the APG TERA. Sampling protocols are delineated in Sections 5 and 6, including sample custody, collection, management, laboratory preparation, and analytical procedures. Section 7 defines data management, and system controls for laboratory data quality are listed in Section 8. Section 9 discusses equipment calibration and maintenance. Sampling and laboratory record keeping are delineated in Section 10. Additional quality control measures are defined in Sections 11-13, and include system and performance audits, corrective action, and quality control reports. Section 14 contains a bibliography of references which was used in the development of this document.

1.1 PURPOSE

Quality assurance (QA) is defined as the overall system of activities for assuring the reliability of data produced. The system integrates the quality planning, assessment, and improvement efforts of various groups in the organization to provide the independent QA program necessary to establish and maintain an effective system for collection and analysis of environmental samples and related activities. The program encompasses the generation of valid and complete data and its subsequent review, validation, and documentation.

1.2 SCOPE

The QAPjP establishes function-specific responsibilities and authorities for data quality and defines procedures which will ensure that field and laboratory activities will result in the generation of quality data. Implementation of the program will ensure the validity of data collected during field and laboratory operations, and establishes sound premises for decision making.

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Inherent in the QA program is the implementation of quality control (QC) measures. These measures provide assurance that the monitoring of quality-related events has occurred, and that data gathered in support of the project is accurate, precise, representative of the sample matrix, and complete.

SECTION 2.0

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2.0 PROJECT DESCRIPTION

2.1 PROJECT OBJECTIVE

The scope of this task is to collect data for a Terrestrial and Ecological Risk Assessment (TERA) of eight study areas within APG, a U.S. Army installation of relatively undeveloped coastal plain uplands, wetlands, and estuary on the upper Chesapeake Bay in Maryland. Since 1918, APG has been used for research, development, and testing of chemical surety materials, conventional munitions, weapons, and other material. Environmental sampling conducted to date has indicated that past testing and waste disposal practices have resulted in chemical contamination in waters, sediment, and soils of APG. More than 700 potential sources of contamination are suspected to exist. Under an interagency agreement (IAG) with USEPA, the Army has agreed to address all potentially affected areas at APG under the CERCLA Remedial Investigation/Feasibility Study (RI/FS) program.

The RI/FS process at APG has been divided into a site characterization portion and a risk assessment portion. Workplans for each portion were developed separately but were designed to complement each other. WES (1991b, 1992), ICF KE (1991), Dames and Moore (1992), and Jacobs Engineering (1991) developed workplans for RI/FS site characterization. ICF KE developed workplans for risk assessment under Task No. 4 (ICF KE 1992) to complement the workplans developed for the RI/FS site characterizations. Under Task No. 10, the work proposed by ICF KE (1992) at eight of the eighteen study areas will be performed in coordination with the work proposed by RI/FS contractors. Preparation of the final human health and ecological risk assessment reports will be included in a subsequent task. This QAPjP was developed to complement the workplans developed under Task No. 4 and includes only a brief summary of information contained in the study area specific workplans included as appendices to the Technical Plan for Risk and Biological Impact Assessment (ICF KE 1992).

2.2 PROJECT SCOPE

The scope of this task is to collect abiotic and biotic data sufficient to characterize the type, magnitude and extent of ecological impact due to contamination in the terrestrial and aqueous habitats associated with the eight study areas of highest priority with respect to risk at APG. All data collected will also be used to support the human health risk assessment.

Collection of data under the TERA will include: (1) conducting screening level field studies to evaluate the type, extent, or existence of biological impacts; (2) conducting studies on chemical accumulation in biological organisms; (3) conducting bioassays to evaluate the toxicity of site contaminants to aquatic and terrestrial wildlife and plants; and, (4) conducting studies to examine the occurrence of histological anomalies in indigenous wildlife populations.

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2.3 SITE HISTORY AND BACKGROUND OF APG

APG is located in southern Harford and southeastern Baltimore counties of Maryland, near the head of the Chesapeake Bay. APG is drained by eight rivers, including the Bush and Gunpowder Rivers. Figure 2-1 is a site location map. The climate at APG is moderated by its proximity to the Chesapeake Bay and the Atlantic Ocean and winters are generally milder than in the inland areas. The area receives approximately 115 cm of rain per year, with maximum precipitation in August and minimum precipitation in October. The mean daily temperatures range from 1°C in winter to 24°C in summer.

Established in 1917 as the Ordnance Proving Ground, APG was designated a formal military post in 1919. Testing of ammunition and material, and operation of training schools began in 1918. Prior to World War II, activities at APG were characterized by intense research and development, and large-scale testing of a wide variety of munitions, weapons, and material. Just before and during World War II, the pace of weapons, munitions, and material testing increased greatly. During the war, personnel strength at APG exceeded 30,000. Similar but small-scale increases in munitions and material development and testing activities at APG were experienced during the Korean and Vietnam conflicts.

From 1940 to 1943, additional land was added to the original APG property. Spesutie Island was acquired in 1945. APG currently houses thirteen major tenant activities, including the U.S. Army Test and Evaluation Command Headquarters and the U.S. Army Ordnance Center and School. Throughout its history, APG's primary mission has been and continues to be the testing and development of weapons, munitions, vehicles, and a wide variety of support material relevant to military operations.

2.4 SITE HISTORY AND BACKGROUND OF ELEVEN STUDY AREAS

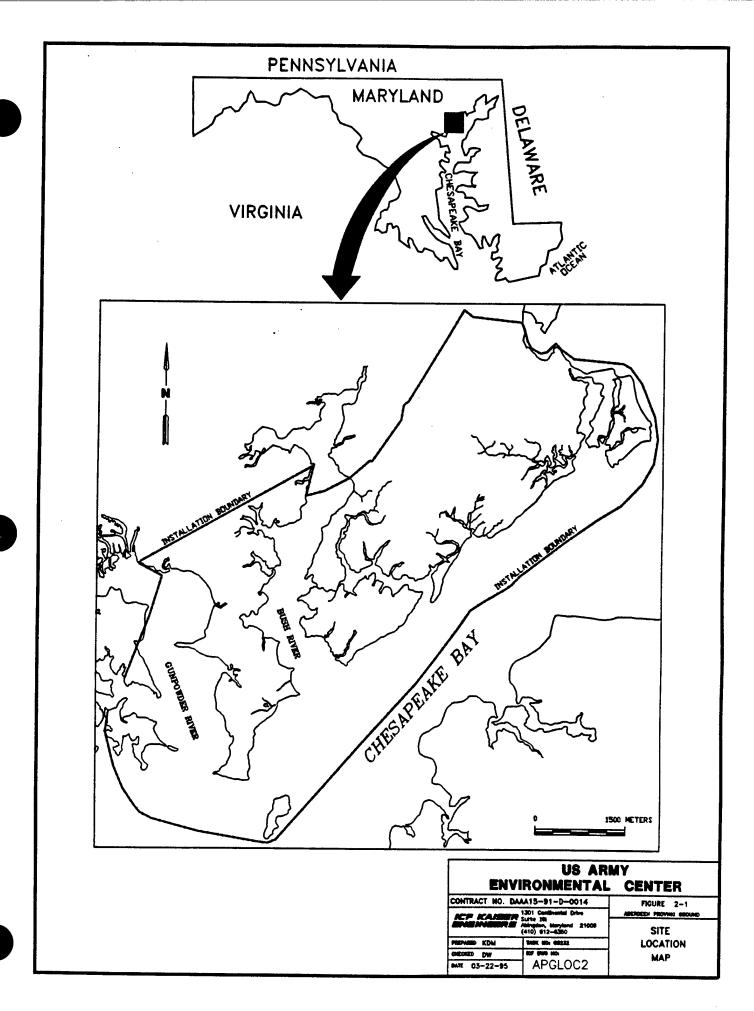
The eleven study areas to be included in the TERA are O-Field, Canal Creek, Cluster 1, Michaelsville Landfill, Bush River, Lauderick Creek, Other Aberdeen Areas, Graces Quarters study areas, Other Creeks Study Area, Other Edgewood Areas, and Western Boundary Study Area. A brief summary of the history and site background of each of the eleven areas is presented below. A more detailed discussion of each of the study areas is provided in ICF KE (1992).

2.4.1 O-Field Study Area

O-Field study area is located within the Edgewood area of APG and covers approximately 105 hectares. It is bordered on the north and east by Watson Creek, on the south by H-Field, and on the west by the Gunpowder River. The O-Field study area contains two disposal areas (Old O-Field and New O-Field) and one suspected disposal area. Past disposal practices at O-Field include the shallow burial or open-pit burning of munitions, solvents, chemical warfare agents, and ordnance. Maps of the O-Field study area are provided in Appendix A of the general Technical Plan (ICF KE 1992).

2.4.2 Canal Creek Study Area

Canal Creek study area is located in the northern section within the Edgewood Area of APG. The study area is defined as the watershed bordered to the north by the Penn Central railroad tracks,



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to the south/southwest by the Gunpowder River, to the south/southeast by a security fence that prevents access to the Gunpowder Neck, to the northeast by Hoadley Road, to the east/southeast by Kings Creek, and to the west by an area just west of the wetlands area of the West Branch of Canal Creek. The Canal Creek drains approximately 1,214 hectares and the Kings Creek drains 324 hectares. Maps of the Canal Creek study area are provided in Appendix C of general Technical Plan (ICF KE 1992).

The Canal Creek study area has been an important chemical warfare research and development center for the United States since 1917. Activities in the Canal Creek study area have included laboratory research, field testing, and pilot-scale and full-scale manufacturing of chemical materials. Portions of the Canal Creek study area also were used for landfilling of sanitary wastes and for the disposal of production wastes. Other activities at the Canal Creek study area included the operation of machine and maintenance shop garages, fabrication of metal parts, degreasing, and metal plating.

2.4.3 Cluster 1 Study Area

Cluster 1 is located in the Edgewood Area of APG and covers an area of approximately 31 hectares. Cluster 1 is one of nine clusters that compose the Lauderick Creek study area and is commonly referred to as the launch area and the barracks area of the Nike Missile Battery. It is bordered to the north by the installation boundary and Amtrack Rail lines, to the southeast by Monks Creek, and to the east, west, and south, by other clusters in the study area. Maps of the Cluster 1 study area are provided in Appendix E1 of general Technical Plan (ICF KE 1992).

The launch area is located at the northern end of Cluster 1 and is surrounded by a fence. It contains six abandoned water-filled missile silos, several other buildings, at least one abandoned underground fuel oil storage tank, and a septic tank with an associated subsurface sand filter bed. The barracks area is located southwest of the launch area. It contains five buildings, a septic tank, and a subsurface sand filter bed. There are also five underground fuel oil storage tanks that were installed in 1957 and are still in use (ANL 1990). Lauderick Creek is located west of the barracks area. Past activities at Cluster 1 include training for chemical warfare and other military activities and installation and maintenance of a Nike missile battery. A variety of chemical compounds, including chemical agents, incendiary materials, solvents, and oils, were associated with these activities.

2.4.4 Michaelsville Landfill Study Area

Michaelsville Landfill is located in the north central portion of the Aberdeen Area of APG. The landfill is situated between Michaelsville Road and Trench Warfare Road in a fenced, controlled area. It is approximately 50 ha in size. Michaelsville Landfill was used from 1969 through 1980, primarily for the disposal of domestic and non-industrial wastes from the Aberdeen Area, but some potentially hazardous wastes also are suspected to have been disposed in the landfill. Maps of the Michaelsville Landfill study area are provided in Appendix J of general Technical Plan (ICF KE 1992).

2.4.5 Bush River Study Area

Bush River study area is located within the Edgewood Area of APG and covers approximately 325 hectares. It is bordered to the north by Lauderick Creek, to the south by Kings Creek, and to the east by Bush River. Lauderick Creek and Kings Creek both drain into the Bush River. Sites within the study area have been used for disposal of chemical agents, munitions, dredge spoil, and radioactive

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materials, by means of burning, dumping, or burial. Other activities included decontamination of one-ton containers of chemicals agents, landfilling, ammunition renovation, chemical storage, dog breeding research, and artillery testing. Maps of the Bush River study area are provided in Appendix D of general Technical Plan (ICF KE 1992).

2.4.6 Lauderick Creek Study Area

Lauderick Creek study area is located within the Edgewood area of APG and covers an area of approximately 31 hectares. It is bordered to the north by the installation boundary and Amtrack Rail lines, to the west by Lauderick Creek, and to the south and east by Bush River. The Lauderick Creek study area is divided into nine clusters. Maps of the Lauderick Creek study area are provided in Appendix E2 of general Technical Plan (ICF KE 1992).

Past activities at these clusters include chemical warfare training and other military training, installation and maintenance of the Nike missile battery, and disposal of wastes related to these two activities. A variety of chemical compounds, including chemical agents, decontaminating agents, incendiary materials, solvents, and oils, were associated with these activities.

2.4.7 Other Aberdeen Areas

An additional thirty-five sites are located on the Aberdeen Area of APG which is surrounded by three large bodies of water: Chesapeake Bay to the east, Swan Creek to the northeast, and Bush River to the west and south. Seven of the 35 areas identified with in the Aberdeen Area by U.S. Army Corps of Engineers Waterways Experiment Station (WES 1990) will be investigated during the APG TERA. The investigation of these seven sites will be supplemented by the overall evaluation of Aberdeen Area through hunting and trapping (harvest) surveys. The seven sites are as follows: Old Dump on Swan Creek, Old Dump on Woodrest Creek, Wastewater Ditch at Shell Washout Facility (Bldg. 700B), Defense Reutilization and Marketing Office (DRMO) Scrap Metal Yard on Michaelsville Road, Old Dump on Spesutie Island, Chemical Dump Ponds on Spesutie Island, and Landfill at Churchville Test Course. Site maps of the seven Other Aberdeen Areas are provided in Appendix N of general Technical Plan (ICF KE 1992).

2.4.8 Graces Quarters Study Area

Graces Quarters is located in the western portion of the Edgewood Area at APG, separated from the main portion of the installation by the Gunpowder River. The Gunpowder River is to the northeast and east of the peninsula, Dundee Creek is to the west, and Saltpeter Creek is to the south. The Hammerman Area of Gunpowder State Park is located to the north of the study area. Graces Quarters is approximately 193 hectares and ranges from sea level to over 12 m above sea level. The primary activities at Graces Quarters were chemical agent and biological simulant testing from the late 1940s to 1971. Maps of Graces Quarters study area are provided in general Technical Plan (ICF KE 1992).

2.4.9 Other Creeks Study Area

The Other Creeks study area consists of seven creeks. Six of the creeks are located on the Aberdeen Area of APG (Woodrest Creek, Swan Creek, Dipper Creek, Mosquito Creek, Spesutie Narrows, and Delph Creek). The seventh creek is located on Spesutie Island (Back Creek). The

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combined area of the watersheds for these creeks covers approximately 5,000 hectares (ha). Very few military-related activities were known to occur on the creeks of this study area, however, a number of activities took place within the watersheds for these creeks. Many of these activities could have caused the influx of contaminates into the creeks. Activities that took place in some of the watersheds included the testing and development of weapons, munitions, vehicles, and a wide variety of support materials used in military operations. Discharges from APG wastewater treatment plants may also have led to the discharge of APG-related contaminants into some of the creeks.

2.4.10 Other Edgewood Study Areas

The Edgewood Areas of APG have been used since 1917 for chemical warfare research and development with activities including laboratory research, field testing of chemical agents, and pilot scale manufacturing. Edgewood Area has also been a center for storage of chemical warfare material and receiving center for waste handling operations including low level radiological waste. During WWI and WWII, Edgewood Area was also the location of production scale chemical agent manufacturing.

The Other Edgewood Study Areas consists of thirty-three clusters, comprised of eighty-four sites, identified during a Resource Conservation and Recovery Act (RCRA) Facility Assessment (RFA) of the Edgewood Area of APG (AEHA 1989). RI/FS work plans have been generated for nine of the clusters comprising Other Edgewood Area as follows: Cluster 4,8,12,16,19,22,23,24, and 25. This QAPjP will address sampling that will be conducted in support of Risk Assessments of these areas.

2.4.11 Western Boundary Area

The Western Boundary Area (WBA) is located in the northwestern corner of the Aberdeen Area of APG and consists of five individual sites including the Aberdeen Fire Training Area (AFTA), the Phillips Army Airfield Landfill (PAAL), the Phillips Army Airfield (PAA), the Test Range for Advanced Aerospace Vulnerability (TRAAV) and the Palmer House Area (PHA). RI/FS investigations have been proposed for these sites to determine the source or sources of the chemical contamination that have been found in the off-post drinking water supply wells. The baseline risk assessment will address these five sites.

2.5 FIELD OPERATIONS

Field operations for the TERA are scheduled to coincide with RI/FS site characterization activities. Schedules for TERA work will be included when site characterization schedules have been finalized. RI/FS site characterization contractors are in communication with ICF KE to coordinate the scheduling of all field operations.

Appendices to the general Technical Plan of the Risk and Biological Impact Assessment for each of the study areas discusses details of work proposed for each of the study areas. Laboratory work to accompany the TERA for each of the study areas is summarized in Tables 2-1 through 2-8. No additional sampling is currently scheduled for the Bush River, Lauderick Creek or Graces Quarters study areas.

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TABLE 2-1
SUMMARY OF LABORATORY BIOASSESSMENT WORK AT O-FIELD STUDY AREA

Species/Analyte	Site Samples	Background	Qua	lity Control Sam	ples			
		Samples	Duplicates ^a	Rinse Blanks ^b	Trip Blanks ^C			
	Surface Soll							
TAL inorganics	20	1	2	1	••			
TCL VOCs	5	1	1	1	1			
TCL semivolatiles, pesticides/PCBs	5	1	1	1				
CSM degradation products	5	1	1	1				
Explosives	5	1	1	1	**			
Dioxin/Furan	2	1	1	1				
		Surface Wa	nter					
Selenastrum capricornutum bioassay	9	1	1					
Water quality	9	1	1					
TAL inorganics	9	1	1					
TCL VOCs	9	1	1	**	1			
TCL semivolatiles, pesticides/PCBs	9	1	1					
CSM degradation products	9	1	1					
Explosives	9	1	1	••				
		Sedimen	t					
Hyalella azteca bioassay	11	1						
TAL inorganics	11	1	2	1				
TCL VOC, semivolatiles, pesticides/PCBs	11	1	2	1				

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TABLE 2-1 (continued) SUMMARY OF LABORATORY BIOASSESSMENT WORK AT O-FIELD STUDY AREA

Species/Analyte	Site Samples	Background	Quality Control Samples			
		Samples	Duplicates ^a	Rinse Blanks ^b	Trip Blanks ^c	
CSM degradation products	11	1	2	1		
Explosives	11	1	2	1	••	
Organomercury	11	1	2	1	**	
USCS, grain Size, TOC, %moisture	11	1			••	
		Benthic Tis	sue			
TAL inorganics	5	1	1	1		
TCL BNAs	5	1	1	1		
CSM degradation products	5	1	1	1		
Explosives	5	1	1	1		

-- A sample will not be collected for analysis.

Duplicates will be collected at a frequency of one per week, per matrix, or 10% of the total number of

Rinse blanks will be collected at a frequency of 5% of number of samples. Benthic tissue samples will be collected in conjunction with sediment samples; therefore the one rinse blank collected will be applicable to both parameters.

One trip blank will be sent with each cooler containing samples for chemical analysis.

BNAs Base Neutral Acid extractables CSM Chemical Surety Material

PCBs Polychlorinated biphenyls

TAL/TCL USEPA's Target Analyte List/Target Compound List

TOC Total organic carbon

USCS United Soil Classification System VOC Volatile Organic Compound

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TABLE 2-2 SUMMARY OF SAMPLES TO BE COLLECTED AT CANAL CREEK STUDY AREA

Site	Species/Analyte	Site	Background	Quality	Quality Control Samples			
		Samples	Samples	Duplicates ^a	licates ^a Rinse Blanks ^b			
			Sediment					
	Hyalella azteca bioassay	4	1	-				
	TAL inorganics	10	2	1	1			
	TCL VOCs	10	2	1	1			
	TCL semivolatiles, pesticides/PCBs	10	2	1	1			
	CSM degradation products	10	2	1	1			
	TCPU	10	2	1	1			
	Explosives	10	2	1	1			
Canal Creek	USCS, grain size, TOC, %moisture	10	2					
	Benthic Tissue ^{e,1}							
	TAL	10	2	1	1			
	TCL pesticides/PCBs	10	2	1	1			
	CSM degradation products	2	2	1	1			
	%lipids	10	2	1	1			
	Organomercury	2	2	1	1			
	Explosives	2	2	1	1			

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TABLE 2-2 (continued) Page 12 of 122 SUMMARY OF LABORATORY BIOASSESSMENT WORK AT CANAL CREEK STUDY AREA

Site	Species/Analyte	Site	Background	Quality	Control San	nples				
		Samples	Samples	Duplicates ^a	Rinse Blanks ^b	Trip Blanks ^c				
	Sediment									
	Daphnia Magna	26	••	**	••					
	TAL inorganics	26	2	3	2					
	TCL VOCs	26	2	3	2					
Kings Creek	TCL semivolatiles, pesticides/PCBs	26	2	3	2	••				
	CSM degradation products	26	2	3	2					
	TCPU	26	2	3	2					
	Explosives	26	2	3	2					
	USCS, grain size, TOC, %moisture	26	2	3	2					

- Duplicates will be collected at a frequency of one per week, per matrix, or 10% of the total number of samples.
- Binse blanks will be collected at a frequency of 5% of number of samples.
- One trip blank will be sent with each cooler containing samples for chemical analysis.
- Storm water refers to surface water collected from Watson Creek and Gunpowder River within 12 hours of a storm event.
- Benthic tissue samples will be collected in conjunction with sediment samples; therefore the one rinse blank collected will be applicable to both parameters.
- f Benthic tissue to be collected include benthic invertebrates and aquatic macrophyte samples.
- A sample will not be collected for analysis.

CSM Chemical surety material PCBs Polychlorinated biphenyls

TAL/TCL USEPA's Target Analyte List/Target Compound List

TOC Total organic carbon

TCPU bis(2,4,6-trichlorophenyl)urea
USCS United Soil Classification System
VOC Volatile organic compounds

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TABLE 2-3 SUMMARY OF SAMPLES TO BE COLLECTED AT CLUSTER 1 STUDY AREA

		Background	Quality Control Samples			
Species/Analyte	Site Samples	Samples	Duplicates ^a	Rinse Blanks ^b		
		Sediment				
Chironomus tentans bioassay	6	1				
TAL inorganics	6	1	1	1		
TCL VOCs	6	1	1	1		
TCL semivolatiles, pesticides/PCBs	6	1	1	1		
USCS, grain size, TOC %moisture	6	1				

Duplicates will be collected at a frequency of one per week, per matrix, or 10% of the total number of samples.

Binse blanks will be collected at a frequency of 5% of number of samples.

A sample will not be collected for analysis.

PCBs Polychlorinated biphenyls TOC Total organic carbon VOC Volatile organic compounds TAL/TCL USEPA's Target Analyte List/Target Compound List USCS United Soil Classification System

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			Quality Control Samples			
Species/ Analyte	Site Samples	Background Samples	Duplicates ^a	Rinse Blanks ^b	Trip Blanks ^c	
		Surface Wate	er			
Rana pipiens bioassay	9	1	. 			
TAL inorganics	3	1	1			
TCL VOCs	3	1	1		1	
TCL semivolatiles	3	1	1	***		
TCL pesticides/PCBs	3	1	1	••		
Explosives	3	1	1			
Water quality	3	1	1			

- No sample will be collected for this analysis.

Duplicates will be collected at a frequency of one per week, per matrix, or 10% of the total number of samples.

Rinse blanks will be collected at a frequency of 5% of number of samples. Benthic tissue samples will be collected in conjunction with sediment samples; therefore the one rinse blank collected will be applicable to both parameters.

One trip blank will be sent with each cooler containing samples for chemical analysis.

TAL/TCL USEPA's Target Analyte List/Target Compound List

PCBs Polychlorinated biphenyls VOC Volatile Organic Compound

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TABLE 2-5 SUMMARY OF SAMPLES TO BE COLLECTED AT BUSH RIVER AREA

	04-		Quality Control Samples					
Species/Analyte	Site Samples	Background Samples	Duplicates	Rinse Blanks	Trip Blanks			
Sediment								
USCS, grain size, TOC, %moisture	TBD	1						

- A sample will not be collected for analysis.

TBD Number of samples collected is to be determined during the initial site survey.

TOC Total organic carbon

USCS United Soil Classification System

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TABLE 2-6 SUMMARY OF SAMPLES TO BE COLLECTED AT OTHER ABERDEEN AREAS

n military and a				Qua	lity Control San	npies			
Site	Species/ Analyte	Site Background Samples Samples		Duplicates ^a	Rinse Blanks ^b	Trip Blanks ^c			
	Sediment								
	Hyalella azteca bioassay	3	*	1		••			
	TAL inorganics	3	*	1	1				
Old Dump on Swan Creek	TCL VOCs, semivolatiles, pesticides/PCBs	3	*	1	1				
	AVS/SEM	3			••				
	Explosives	3		1	1				
	USCS, grain size, TOC, %moisture	1							
	Sediment								
	Hyalella azteca bioassay	3	*	1		••			
	TAL inorganics	3	*	1	1				
Old Dump on Woodrest	TCL VOCs, semivolatiles, pesticides/PCBs	3	*	1	1				
Creek	AVS/SEM	3		••		••			
	Explosives	3		1	1				
	USCS, grain size, TOC, %moisture	1							
			Sediment						
	Hyalella azteca bioassay	3	*	1	••				
Mactawatar	TAL inorganics	3	*	1	1				
Wastewater Ditch at Shell Washout	TCL VOCs, semivolatiles, pesticides/PCBs	3	*	1	1	••			
Facility	AVS/SEM	3			**				
	Explosives	3	••	1	1	**			
	USCS, grain size, TOC, %moisture	1			**				

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TABLE 2-6 (continued) SUMMARY OF LABORATORY BIOASSESSMENT WORK AT OTHER ABERDEEN AREAS

Site	Species/ Analyte	Site Background Samples Samples*	Quality Control Samples					
				Duplicates ^a	Rinse Blanks ^b	Trip Blanks ^c		
	Sediment							
	Hyalella azteca bioassay	2	*	1	4-			
	TAL inorganics	2	*	1	**			
DRMO Scrap Metal Yard	TCL VOCs, semivolatiles, pesticides/PCBs	2	*	1				
Wioldi Taio	AVS/SEM	2		••				
	Explosives	2		1		••		
	USCS, grain size, TOC, %moisture	1						

- Duplicates will be collected at a frequency of one per week, per matrix, or 10% of the total number of samples.
- Binse blanks will be collected at a frequency of 5% of number of samples.
- One trip blank will be sent with each cooler containing samples for chemical analysis.
- A sample will not be collected for analysis.
- * Samples will be compared with the Reference Sampling Program.

AVS/SEM Acid Volatile Sulfides/Simultaneously Extracted Metals

PCBs Polychlorinated biphenyls

TAL/TCL USEPA's Target Analyte List/Target Compound List

TOC Total organic carbon

SVOCs Semivolatile Organic Compounds
USCS United Soil Classification System
VOC Volatile Organic Compounds

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TABLE 2-7 SUMMARY OF SAMPLES TO BE COLLECTED AT WESTERN BOUNDARY AREA

Site	Species/ Analyte			Qua	Quality Control Samples			
			Background Samples	Duplicates ^a	Rinse Blanks ^b	Trip Blanks ^c		
Sediment								
	TAL inorganics	5	*	1	1			
	TCL VOCs, semivolatiles, pesticides/PCBs	5	*	1	1			
Fire Training	Explosives	5		1	1			
Area	Surface Water							
	TAL inorganics	5	*	1	1	1		
	TCL VOCs, semivolatiles, pesticides/PCBs	5	*	1	1			
	Explosives	5		1	1			

- Duplicates will be collected at a frequency of one per week, per matrix, or 10% of the total number of samples.
- Brinse blanks will be collected at a frequency of 5% of number of samples.
- One trip blank will sent with each cooler containing samples for chemical analysis.
- A sample will not be collected for analysis.
- * Samples will be compared with the Reference Sampling Program.

AVS/SEM Acid Volatile Sulfides/Simultaneously Extracted Metals

PCBs Polychlorinated biphenyls

TAL/TCL USEPA's Target Analyte List/Target Compound List

TOC Total organic carbon

SVOCs Semivolatile Organic Compounds
USCS United Soil Classification System
VOC Volatile Organic Compounds

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TABLE 2-8 SUMMARY OF SAMPLES TO BE COLLECTED AT OTHER CREEKS AREA

Site	Species/ Analyte			Quality Control Samples			
			Background Samples	Duplicates ^a	Rinse Blanks ^b	Trip Blanks ^c	
Dipper Creek	Sediment						
Swan Creek Spesutie	TAL inorganics	26	*	3	1		
Narrows Back Creek Woodrest Creek	TCL VOCs, SVOCs, pesticides/PCBs	26	*	3	1		
	Explosives	26		3	1		

- Duplicates will be collected at a frequency of one per week, per matrix, or 10% of the total number of samples.
- Plinse blanks will be collected at a frequency of 5% of number of samples.
- One trip blank will sent with each cooler containing samples for chemical analysis.
- A sample will not be collected for analysis.
- * Samples will be compared with the Reference Sampling Program.

AVS/SEM Acid Volatile Sulfides/Simultaneously Extracted Metals

PCBs Polychlorinated biphenyls

TAL/TCL USEPA's Target Analyte List/Target Compound List

TOC Total organic carbon

SVOCs Semivolatile Organic Compounds
USCS United Soil Classification System
VOC Volatile Organic Compounds

SECTION 3.0

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3.0 PROJECT ORGANIZATION AND QA/QC RESPONSIBILITY

Quality assurance goals for the TERA will be achieved through proper planning, organization, review, communication of objectives, auditing, reporting, and corrective action. The QA program will be carried out by personnel knowledgeable in QA theory and practice. Facilities, equipment, and services which affect data quality or integrity will be routinely inspected and maintained, as required by SOPs.

Implementation of the QAPjP requires that the project staff maintain an awareness of contractual procedures and goals. It is the policy of ICF KE to provide a QA program to ensure that all information produced by its employees and subcontractors is valid and of known quality. QA program requirements cover all activities which generate environmental measurement data. These requirements include statements of completeness, comparability, representativeness, precision, and accuracy, where applicable.

Program personnel will be familiar with the required conventions, formats, and schedules specified in documents pertinent to project activities. Data review personnel will review data for accuracy and precision in accordance with USEPA Region III guidelines. ICF KE Statement of Qualifications is provided in Appendix B.

Field methods and procedures used in measurement and monitoring efforts will conform to USEPA- and USAEC-approved methodologies, where applicable. Field team members will possess the appropriate qualifications and training prior to collecting environmental samples and performing geology-related tasks. All measurement methods will be fully documented and will include quality control procedures.

The intended use(s) of the data and the associated acceptance criteria for data quality will be determined before the data collection effort begins. Reported data will include, when appropriate, statements of precision, accuracy, representativeness, completeness, and comparability. Data processing procedures will be documented, reviewed, and revised, as required to meet USAEC and USEPA data quality requirements.

3.1 PROJECT ORGANIZATION

The Commander of USAEC is ultimately responsible for the data collected in support of Agency projects. The Commander delegates the applicable authority to the Contracting Officer's Representative (COR) who delegates authority to the Project Officers. John Paul has been designated the Project Officer for the APG TERA. His responsibilities include:

- a. Overseeing and monitoring performance of all TERA participants;
- b. Interfacing with regulatory agencies;
- c. Liaison between USAEC and ICF KE;
- d. Requiring effective implementation of the USAEC QA Program; and

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e. Requiring completion of corrective actions, when indicated.

Figure 3-1 denotes the line of authority and project organization for the APG TERA. Principal project participants and responsibilities for the project QA program reside primarily in personnel from ICF KE. Table 3-1 provides the names, titles, addresses and phone numbers of personnel responsible for ensuring the maintenance of the APG Quality Assurance Program.

3.2 RESPONSIBILITIES

3.2.1 Project QA/QC

Responsibilities for implementation of the project QA program in accordance with QA/QC contractual obligations lies principally with the Project Task Manager and the QA Manager. The Project Task Manager delegates to the QA Manager the authority to ensure the reliability and validity of project activities and deliverables in compliance with the project QA program and the USEPA Region III guidelines. The QA Manager or the designated QA Officer, has specific responsibilities which include the following:

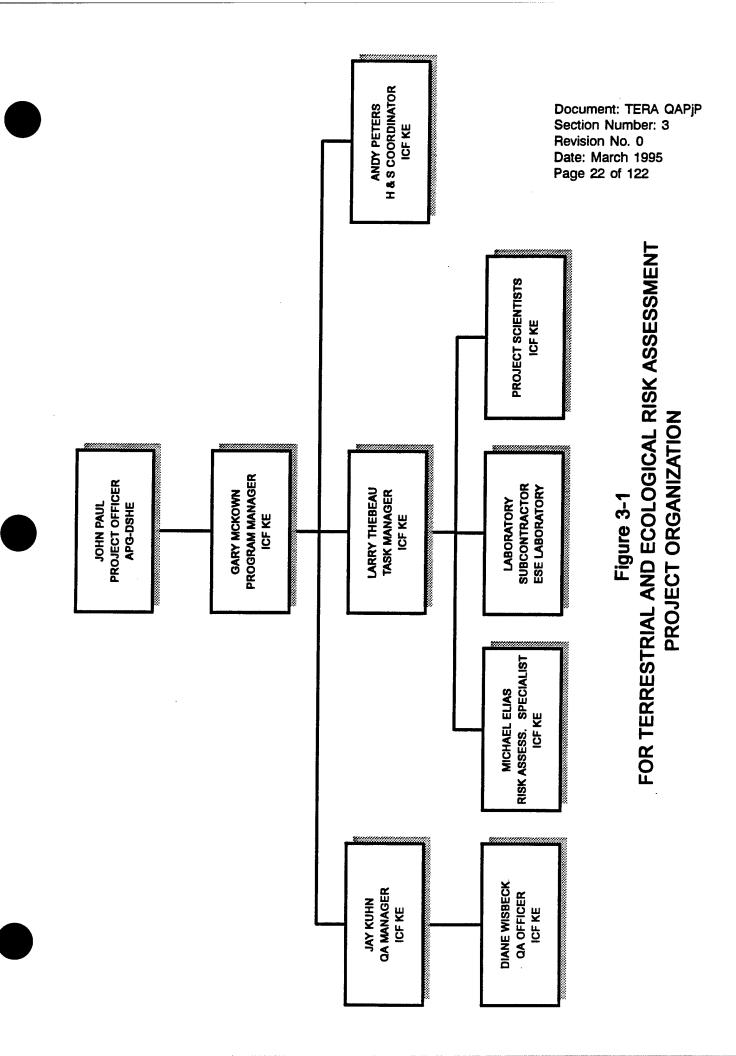
- Initiating QA activities within the program to ensure that QC measures are being implemented and maintained;
- b. Ensuring all records, logs, SOPs, and analytical results are documented and maintained in a retrievable manner;
- c. Conducting periodic performance surveys to ensure acceptable analytical performance;
- d. Preparing periodic quality reports, and QA sections of final reports; and
- e. Ensuring corrective action has been implemented and documented to preclude future occurrences.

The QA Manager and the QA Officer have the responsibility and the authority to report directly to the Program Manager, USAEC Project Officer, and the laboratory, as necessary, to resolve any conflicts deemed adverse to quality achievement.

3.2.2 Field Activities

Responsibilities for implementation of the QA program in conjunction with field activities lies principally with the Field Operations Leader. The Field Operations Leader will ensure that all field team members possess appropriate qualifications and training prior to collecting environmental samples and performing geology-related tasks. Specific responsibilities include the following:

- Ensuring that sampling activities are consistent with the approach defined in this QAPjP;
- b. Ensuring that QC measures are being implemented and maintained;



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TABLE 3-1 KEY INDIVIDUALS FOR THE TERA

NAME	TITLE 11 1 1	ADDRESS	PHONE NUMBER
John Paul	Project Officer	Directorate of Safety, Health, and the Environment (DSHE) Aberdeen Proving Ground, MD 21010-5401	(301) 671-4840
Gary McKown	Program Manager ICF Kaiser Engineers	1301 Continental Drive Ste. 101 Abingdon, MD 21009	(410) 612-6358
Jay Kuhn	Quality Assurance Manager	1301 Continental Drive Ste. 101 Abingdon, MD 21009	(410) 612-6369
Larry Thebeau	Task Manager ICF Kaiser Engineers	1301 Continental Drive Ste. 101 Abingdon, MD 21009	(410) 612-6357
Diane Wisbeck	Quality Assurance Officer ICF Kaiser Engineers	1301 Continental Drive Ste. 101 Abingdon, MD 21009	(410) 612-6361
Mike Lowe	Equipment Manager ICF Kaiser Engineers	9300 Lee Hwy. Fairfax, VA 22031-1207	(703) 934-3621
Kim Mason	Project Data Coordinator ICF Kaiser Engineers	1301 Continental Drive Ste. 101 Abingdon, MD 21009	(410) 612-6372
Andy Peters	Health and Safety Officer ICF Kaiser Engineers	9300 Lee Hwy. Fairfax, VA 22031-1207	(703) 934-3887

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- c. Ensuring that all records, and logs, are documented and maintained in a retrievable manner; and
- d. Elucidating conditions requiring corrective actions and implementing the appropriate course of action.

3.2.3 <u>Laboratory Activities</u>

The laboratory chosen to provide chemical analytical support for the TERA is Environmental Science and Engineering (ESE). The laboratory selected to perform physical analyses is ICF KE Laboratory. The analytical task managers of ESE and ICF KE will be responsible for maintaining quality assurance of the respective laboratories. Responsibilities include the following: (1) to provide sufficient equipment, space, resources, and personnel to conduct analyses and implement the QA program; (2) to submit the required documented methods and laboratory certification prior to analyzing samples; (3) to ensure that sampling and other handling procedures are adequate for the sample types received; (4) to oversee the quality of purchased laboratory materials, reagents, and chemicals to ensure that these supplies do not jeopardize the quality of analytical results; and (5) to ensure the implementation of corrective action for any QA/QC deficiencies. In addition to these requirements, the task manager of ESE will be required to ensure the integrity of the analyses performed by IT Laboratory, subcontracted by ESE to perform dioxin/furan analyses, in accordance with ESE's Quality Assurance Manual (QAM). ESE Laboratory's Statement of Qualification (SOQ) and QAM are provided in Appendix E. IT Laboratory's QAM is provided in Appendix F.

The specific auditing functions include:

- a. Monitoring laboratory QA/QC functions to ensure that practices are in conformance with approved policies and SOPs, and that quality controls indicate acceptable data quality;
- b. Evaluating QA activities within the program to ensure that QC measures are being implemented and maintained;
- c. Ensuring that all records, logs, SOPs, and analytical results are maintained in a retrievable manner;
- d. Elucidating conditions requiring corrective actions and implementing the appropriate course of action; and
- e. Conducting periodic performance and system audits to ensure acceptable analytical performance.

In addition, USAEC evaluates the QA/QC of analytical methods on a real-time basis by reviewing weekly control chart submittals, and evaluating weekly quality control reports for non-conformance and corrective action activities. Protocols for the analysis of samples using USAEC-certified methods are contained in the USATHAMA QA program (1990), which are based on USEPA methods.

SECTION 4.0

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4.0 QUALITY ASSURANCE AND DATA QUALITY OBJECTIVES

4.1 QUALITY ASSURANCE

The overall QA objective is to develop and implement procedures for sample and data collection, sample shipment, and reporting that will allow QA reviewers to determine, with reasonable certainty, whether the field and laboratory data collected during the APG TERA meet the criteria and endpoints established in the Data Quality Objectives (DQOs). The QA objective will be achieved through the implementation of specific procedures for sampling, field data collection, chain-of-custody, calibration, internal quality control, audits, preventive maintenance and corrective actions as described in this QAPjP.

4.2 DATA QUALITY INDICATORS

Data will be evaluated using the data quality indicators; accuracy, precision, representativeness, completeness, and comparability. These terms will be applied to both laboratory and field quality assurance procedures. This section defines each data quality indicator and the criteria established for each for this project. Table 4-1, 4-2 and 4-3 summarize aspects of the QA program and correlate the indicators they affect.

4.2.1 Accuracy

Accuracy is the degree of agreement of a measurement (or an average of measurements of the same thing), X, with an accepted reference or true value, T, usually expressed as the difference between the two values, X-T, or the difference as a percentage of the reference or true value, 100 (X-T)/T, and sometimes expressed as a ratio, X/T. Accuracy is a measure of bias in a system.

Sampling accuracy will be quantitatively assessed through the evaluation of trip blank and rinse blank data. This information indicates whether or not contamination has been introduced during the sampling event or during sample transit. The rinse blank data will be collected at five percent of the total number of samples per media (where applicable), and will provide an assessment of decontamination efficiency and the potential for cross-contamination to occur during the field investigation.

Accuracy in the analytical laboratory will be assessed through the evaluation of the percent recoveries associated with reference samples (i.e., matrix spikes, surrogates, continuing calibration checks). Potential sample contamination contributed by the laboratory environment will be discerned through the evaluation of the laboratory method blanks. Method blanks will be processed at the beginning of each analytical run by the laboratory to evaluate whether the internal laboratory environment, reagents used during analyses, analytical techniques, or the instrumentation system are sources of contamination that could affect the integrity of the sample.

The criterion for evaluating blank contamination applies to any blank (field and laboratory) associated with the samples and dictates that no contamination should be found in the blank. If contamination is detected, all data associated with the blank will be evaluated to determine if there is an inherent variability in the data for the lot, or if the problem is an isolated occurrence not affecting all samples in the lot. In cases where more than one blank is associated with a given sample,

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TABLE 4-1 USAEC ANALYTICAL QC ELEMENTS OF QA PROGRAM

tem	DQOª	Parameter	Frequency of Association	Criteria Requirement		
	LABORATORY QC CHECKS					
Analytical Method	С	Explosives; CSM degradation products	each analysis	Approved USAEC SOP		
USAEC Chemical Data Packages	A,P,C	Explosives; CSM degradation products	each lot	Pass ESE peer review and formal QA/QC check; pass USAEC QA/QC check		
Quarterly Laboratory Internal Audit Reports	R	Explosives; CSM degradation products	quarterly	No deficiencies		
Laboratory Chain of Custody	R	Explosives; CSM degradation products	all sample containers	No deficiencies		
Laboratory System Controls	R	Explosives; CSM degradation products	during laboratory operations	Custody of sample within laboratory fully accounted for and documented		
Holding Time	A,P,R	Explosives; CSM degradation products	each analysis	No deficiencies as determined by CEMRD/USAEC approved laboratory QAPP		
USAEC Calibration Method Detection Limit	A,P,R	Explosives; CSM degradation products	each analysis; each calibration	USAEC derived MDL; demonstrated with each initial calibration		
USAEC Calibration - Upper Reporting Limit	A,P,R	Explosives; CSM degradation products	each analysis; each calibration	Linear range of analysis; demonstrated with each initial calibration		
Standard Matrix Method Blank	R	Explosives; CSM degradation products	each analysis	Clean except for common lab contamination		
Standard Matrix Spikes	A,P	Explosives; CSM degradation products	3 per lot	Acceptable recovery as determined by control charts defined in the QAPP.		
Surrogates	A	Explosives; CSM degradation products	all samples if required by method	Acceptable recovery as determined by control windows which will be defined in the QAPP.		

^a P=Precision, A=Accuracy, R=Representativeness, C=Comparability

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TABLE 4-2 USEPA QC ELEMENTS OF QA PROGRAM

tem	DQO®	Parameter	Frequency of Association	Criteria Requirements
Analytical Method	С	TAL/TCL, D/F ^b	each analysis	Certfied USAEPA CLP method
USEPA CLP like data packages	A,P,C	TAL/TCL, D/F	1 per SDG ^c	Pass ESE peer review and formal QA/QC check. Undergo rigorous validation by QC Manager
Prime Contractor Audits	R	TAL/TCL, D/F	as needed	Identified deficiencies are corrected
Laboratory Chain-of- Custody	R	TAL/TCL, D/F	all sample containers	Custody of sample within laboratory full accounted for and documented
Laboratory control checks ^d	R	TAL/TCL, D/F	1 per SDG	As given in method
USEPA CRQL/CRDL	A,P	TAL/TCL, D/F	per calibration	Linear range of analysis to include SQL
Method Blank	Α	TAL/TCL, D/F	1 per SDG	No contamination detected
Sample matrix Spike and duplicates	P	TAL/TCL, D/F	1 per SDG	As given in analytical method
Surrogates	A	TCL	all samples as required by method	As given in analytical method

^a P=Precision, A=Accuracy, R=Representativeness, C=Comparability

b Target Analyte List/Target Compound List, Dioxin/Furan

c SDG= Sample delivery group

Laboratory QC checks include continuing calibration verification standards and blanks as well as laboratory replicate samples.

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TABLE 4-3 FIELD QC ELEMENTS OF QA PROGRAM

item	DQOª	Parameter	Frequency of Association	Criteria Requirement
Source Water	R	ali	per site	Less than USAEC MDL or if detected approved by USAEC
Field Duplicates	P	ali	1 per 20 samples	Water 3X difference; Soil 5X difference
Trip Blank	R	Volatiles in water	1 per cooler with volatiles	Clean
Rinse Blank	R	all	1 per 20 samples	Clean
Chain of Custody	R	all	every sample	Filled out correctly to include signatures; no missing or incorrect information
Field Parameter Forms	R	all	every sample	Filled out correctly to include analytical parameters; map file data; and applicable IRDMIS coding information.
Field Instrument Calibration Logs	A	all	every measurement	All measurements must have associated calibration reference.

^a P=Precision, A=Accuracy, R=Representativeness, C=Comparability

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evaluation will be based upon a comparison with the associated blank having the highest concentration of the contaminant.

Accuracy of field measurements will be qualitatively controlled through the use of SOPs which have been developed to standardize the collection of measurements and samples. Consistent proper calibration of all equipment throughout the field exercises, as described in this QAPjP, will assist in the accuracy of measurements. Field documentation and QA audits will be used to establish that protocols for sampling and measurement follow appropriate SOPs.

4.2.2 Precision

Precision refers to the level of agreement among repeated measurements of the same parameter. It is usually stated in terms of standard deviation, relative standard deviation, relative percent difference, range, or relative range. The overall precision of a piece of data is a mixture of sampling and analytical factors. The analytical precision is easier to control and quantify because the laboratory is a controlled, and therefore, measurable environment. Sampling precision is unique to each site, making it harder to control and quantify. The goals for each factor will be addressed here separately.

For samples collected for chemical analysis, sampling precision will be checked by obtaining one duplicate sample for every 10 samples collected for each type of media (10%). Precision will be evaluated by calculating the relative percent difference (RPD) as follows:

$$RPD = \frac{(XA - XB)}{XM} \times 100$$

XA > XB

XA and XB are duplicate analyses, and XM is the mean value of duplicate analysis XA and XB

The RPD will be calculated for each analytical parameter. For aqueous matrices, those analytes having a RPD greater than 25% will be identified and result in an investigation of the sampling process in order to identify any deviations and if necessary use corrective action to restore proper functioning to the system. For solid matrices, those analytes having an RPD > 50% will be identified and result in an investigation in order to identify any deviations, and if necessary use corrective action to restore proper functioning to the system. Note, information from a single pair of duplicates presents an inaccurate estimate of precision; therefore, evaluation of the overall sampling precision can not be made based on the assessment of a single duplicate pair RPD.

Laboratory precision will be addressed through the analysis of laboratory replicate samples by the contract laboratory. The RPD for each analytical parameter will be calculated as a measurement of precision. The National Functional Guidelines for Inorganics RPD standard of ≤20% for water duplicates will be adopted as the criteria that the inorganic duplicates must meet. If these criteria are not met, an examination of the data similar to that described above will be conducted to determine the cause of the variability and usefulness of the data.

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4.2.3 Representativeness

Representativeness is a measure of the degree to which the measured results accurately reflect the medium being sampled. It is a qualitative parameter which is addressed through the proper design of the sampling program in terms of sample location, number of samples, and actual material collected as a "sample" of the whole.

Sampling protocols have been developed to assure that samples collected are representative of the media. Field handling protocols (e.g., storage, handling in the field, and shipping) have also been designed to protect the representativeness of the collected samples. Proper field documentation and QA audits will be used to establish that protocols have been followed and that sample identification and integrity have been maintained.

4.2.4 Completeness

Completeness is a measure of the amount of information that must be collected during the field investigation to allow for a successful achievement of the objectives. A certain amount and type of data must be collected for conclusions to be valid. Missing data may reduce the precision of estimates or introduce bias, thus lowering the confidence level of the conclusions. While completeness has been historically presented as a percentage of the data that is considered valid, this does not take into account critical sample locations or critical analytical parameters.

The amount and type of data that may be lost due to sampling or analytical error cannot be predicted or evaluated in advance. The importance of any lost or suspect data will be evaluated in terms of the sample location, analytical parameter, nature of the problem, decision to be made, and the consequence of an erroneous decision. Critical locations or parameters for which data is determined to be inadequate may be resampled. For this project, the completeness criterion is set in the range between 80-100 percent. It will be assumed that a high degree of data completeness will be obtained.

4.2.5 Comparability

Comparability is the confidence with which one data set can be compared to another. Comparability will be controlled through the use of SOPs which have been developed to standardize the collection of measurements and samples. Consistent proper calibration of all equipment throughout the field exercises, as described in this QAPjP, will assist in the comparability of measurements. Field documentation and QA audits will be used to establish that protocols for sampling and measurement follow appropriate SOPs.

4.3 DATA QUALITY OBJECTIVES

Data Quality Objectives (DQOs) are qualitative and quantitative statements which outline the decision making process and specify the data required to support the APG TERA. DQOs are statements of the level of uncertainty that will be accepted in results derived from environmental data. DQOs defined for this project are provided in tabular format by study area as Tables 4-3 through 4-13. The following factors considered in determining these DQOs include:

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- a. the purpose of collecting data associated with each field activity;
- b. the data types required to meet the objectives (included is the estimated number of data or samples that will be collected to meet the data objective);
- c. the sampling method being employed for each type of data;
- d. the use(s) for which data are being collected (described by using general purpose categories which represent different data uses (e.g., site characterization, risk assessment));
- e. the identification of an appropriate analytical level for the analysis (or measurement) being performed. Five such levels have been defined by USEPA (USPEA 1987);
- f. the analytical method that will be employed to analyze samples;
- g. the typical detection limit requirements for the chosen analytical methods;
- h. evaluation of critical samples, those for which valid data must be obtained to satisfy the objectives of the sampling and analysis task; and
- i. the types and numbers of quality control samples that will be collected in association with each sampling event/media.

TA DATA QUALITY OBJECTIV

DQO	Soil Investigation	Surface Water Investigation	
Objective	Earthworm assays will be performed as an initial screening on soil samples (41) collected from the O-Field study area to evaluate the potential for soil contaminants to impact to terrestrial species.	Bioassays will be performed on surface water collected from Canal Creek and the Gunpowder River in order to evaluate potential impacts of surface water contaminants to aquatic species at a variety of trophic levels. Chronic bioassays will be performed with Selenastrum capricomutum a phytoplankton indigeneous to the region (USEPA 1989). Abiotic chemical analyses-used to support bioassay data	Sediment impacts of Sampling Gunpowo Chronic to the fall of (Nebeker Ablotic of data.
Bioassay endpoint	Earthworm lethality Impact will be indicated by a statistically significant reduction in survival	Impacts on aquatic tissue will be indicated by a statistically significant reduction in survival, reproduction, or fecundity in organisms exposed to creek surface water.	Impacts of statistical fecundity
Chemical data	Soil samples: Subset analyzed for TAL/TCL, CSM degradation products, explosives, and dioxin/furans	Surface water samples: TAL/TCL, CSM degradation products, explosives, water quality Reference samples: TAL/TCL and water quality	Sedimen explosive Reference
Physical data	Map coordinates of the sampling locations will be generated.	pH, temperature, condition, DO, salinity, alkalinity, and hardness Map coordinates of the sampling locations will be generated.	USCS, g Map coo
Sampling method	Environmental, biased, grab, and intrusive	Environmental, biased, composite, intrusive	Environm
Data use	Risk assessment	Risk assessment	Risk ass
Data Level	Earthworm Data: Level II Chemical Data: Level III Physical Data: Level I	Chemical Data: Level III Physical Data: Level I Bioassay Data: Level II	Chemica Bioassay Physical Other Ph
Chemical analytical method	Not applicable	USEPA CLP, USEPA SW-846, USAEC, Standard Methods	USEPA (
Typical detection	Not applicable	Approximately 1 g/L	Approxim
Quality control samples	Not applicable	Trip blanks, duplicates, rinse blanks	Duplicat
Background samples	Bioassay reference samples: Surface soil samples will be collected from a location defined by the background task (ICF KE 1992).	Abiotic data: Background sampling task (ICF KE 1992) Bioassay reference samples: Surface water samples will be collected from Watson Creek and from the Gunpowder River.	Abiotic c Bioassay Swaderic Creek.

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TABLE 4-4
TIVES FOR O-FIELD STUDY AREA

Sediment Investigation	Tissue Residue Analysis	Thiodiglycol Bioassay
ent bloassays will be performed to evaluate potential s of sediment contaminants to benthic invertebrates. ng locations include Watson Creek and the wder River.	Five composite aquatic macrophyte samples will be collected for residue analysis to evaluate the potential accumulation of chemicals in the aquatic food web.	Toxicity of thiodiglycol to freshwater and estuarine aquatic invertebrates will be evaluated using clean surface water spiked with 7 different concentrations of thiodiglycol: 200,000 g/L, 100,000 g/l, 50,000 g/L, 25,000 g/L, 12,500 g/L, 6,250 g/L, and 3,125 g/L.
c toxicity tests will be conducted with <i>Hyalella azteca</i> in on sample locations having no acute toxicity in spring (er et al. 1984).		
: chemical analyses will be used to support bloassay	·	
s on aquatic invertebrate will be indicated by a cally significant reduction in survival, reproduction, or	Not applicable	Mysidopsis bahla: Chronic-survival, reproduction, and fecundity (7 days)
lity in organisms exposed to creek sediment.		Daphnia magna: Chronic-survival and reproduction (21 days)
ent samples: TAL/TCL, CSM degradation products, ives, and organomercuric compounds	Tissue samples: TAL, TCL BNAs, CSM degradation products, and explosives	None
:nce sample: TAL/TCL and organomercuric compounds	Reference samples: TAL/TCL and organomercuric compounds	
, grain size, TOC, %moisture	Field identification of species	None
pordinates of the sampling locations will be generated.	Map coordinates of the sampling locations will be generated.	
nmental, blased, composite, and intrusive	Environmental, blases, composite, and intrusive	No sampling will be performed.
ssessment	Risk assessment	Risk assessment
ical Data: Level III say Data: Level III :al Analytical: Level III Physical Data: Level I	Chemical Data: Level III Physical Data: Level I	Bioassay Data: Level II
A CLP, USEPA SW-846, USAEC	USEPA CLP, USEPA SW-846, USAEC	Not applicable
ximately 1 mg/kg	Approximately 1 mg/kg	Not applicable .
cates and rinse blanks	Duplicates and rinse blanks	Not applicable
c data: Background sampling task (ICF KE 1992)	Biotic data: locations to be defined by the background task (ICF KE 1992)	Not applicable
say reference sample will be collected from either lerick Creek, Dundee Creek, Seneca Creek, or Saltpeter c.		

TABLE 4 DATA QUALITY OBJECTIVES FOR

DQO		CANAL CREEK INVESTIGATION	
	Site Survey	Sediment Investigation	Tissue Residue Anaf
Objective	A site survey will be performed to identify habitat types and potential receptor species in Canal Creek area.	Sediment assays will be performed to evaluate potential impacts of sediment contaminants to benthic invertebrates. Sampling locations include freshwater locations (upper Canal Creek) and estuarine locations (Gunpowder River and lower portions of Canal Creek). Assay to be performed include Chironomus tentans and Hyalella azteca (Nebeker et al., 1984). Abiotic chemical analyses will be used to support bloassay data.	Benthic invertebrate and aquatic m samples will be collected for whole analysis to evaluate the potential for be accumulated in the aquatic food
Bioassay endpoint	Not applicable	Impact on receptor species will be indicated by a statistically significant reduction in survival or growth in organisms exposed to sediment collected from Canal Creek and Gunpowder River relative to reference sediment.	Not applicable.
Chemical data	None	Sediment samles: TAL/TCL, CSM degradation products, explosives, and organomercuric compounds.	Tissue: TAL/TCL BNAs, CSM degration TCPU, organomercuric compounds
		Reference samples: TAL/TCL and organomercuric compounds.	Reference samples: TAL/TCL and compounds.
Physical data	A habitat map will be generated. pH, temp, cond, DO, and salinity.	Map coordinates of the sampling locations will be generated.	Map coordinates of the sampling lo generated.
		Physical testing: grain size, USCS, %moisture, TOC.	Identification of species.
Sampling method	Environmental, nonbiased, grab, Intrusive	Environmental, blased, composite, and intrusive.	Environmental, blased, composite,
Data use	Risk Assessment	Risk Assessment	Risk Assessment
Data level	Physical Data: Level I	Chemical Data: Level IV Bioassay Data: Level II Physical Analytical: Level IV Other Physical Data: Level I	Chemical Data: Level IV Physical Data: Level I
Chemical analytical method	Not applicable	USEPA CLP, USEPA SW-846, USAEC, Standard Methods, ASTM	USEPA CLP, USEPA SW-846, USAI Methods, ASTM

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3LE 4-5 FOR CANAL CREEK STUDY AREA

	KINGS	TCPU BIOASSAY	
e Analysis	Site Survey	Sediment Investigation	
iatic macrophyte whole body residue ntial for chemicals to ic food web.	A site survey will be performed to identify habitat types and potential receptor species in Kings Creek area.	Microtox assays will be performed as an initial screening on sediment samples (40) collected from Kings Creek to evaluate the potential for impact to receptor species. Abiotic chemical analyses will be used to support bioassay data.	Toxicity of TCPU to freshwater and estuarine benthic invertebrates will be evaluated using clean sediment spiked with 5 different concentrations of TCPU (80%, 60%, 40%, 20%, and maximum concentration found in sediment samples.
	Not applicable	Microtox assay: Bacterial luminescence An impact will evaluated by a statistically significant difference in results of sediment collected from Canal Creek and Gunpowder River relative to reference sediment.	Chironomus tentans: survival (10 days) Hyalella azteca: survival (28 days)
I degradation products, ounds and explosives.	Not applicable	Sediment samples: TAL/TCL, CSM degradation, organomercuric compounds	None
L and organomercuric		Reference samples: TAL/TCL, organomercuric compounds	
aling locations will be	A habitat map will be generated. pH, cond., temp., and salinity	Map coordinates of sampling locations will be generated. Physical testing: grain size, USCS, %solids, TOC	None
osite, and intrusive.	Environmental, nonblased, grab, and intrusive	Environmental, biased, grab, and intrusive	No sampling will be performed.
	Risk Assessment	Risk Assessment	Risk Assessment
	Physical Data: Level I	Microtox Data: Level I Chemical Data: Level IV Physical Analytical: Level V Other Physical: Level I	Bioassay Data: Level II
, USAEC, Standard	Not applicable	USEPA CLP, USEPA SW-846, USAEC, Standard Methods	Not applicable

TABLE 4-5 (c DATA QUALITY OBJECTIVES FOF

DQO	CANAL CREEK INVESTIGATION			
	Site Survey	Sediment Investigation	Tissue Realdue Ar	
Typical detection limit	Not applicable	Approximately 1 mg/kg.	Approximately 1 mg/kg.	
Quality control samples	None	Duplicates and rinse blanks	Duplicates and rinse blanks	
Background samples	A similar habitat will be located as part of background sampling task (ICF KE 1992).	Ablotic data: Background sampling task (ICF KE 1992). Bioassay reference samples: Two surface water samples will be taken at a location defined by the background task (ICF KE 1992).	Biotic data: Collected at 2 location background task (ICF KE 1992).	

VAs--Base Neutral Acids Cond.--Conductivity CSM--Chemical Surety Materials DO--Dissolved Oxygen TAL/TCL--Target Analyte/Target Compound List TCPU--bls(2,4,6-tric

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5 (continued) OR CANAL CREEK STUDY AREA

	KINGS CREEK INVESTIGATION		TCPU BIOASSAY
s Analysis	Site Survey	Sediment Investigation	
. ,	Not applicable	Approximately 1 mg/kg	Not applicable
	Not applicable	Duplicates and rinse blanks	Not applicable
cations defined by the 92).	A similar habitat will be located as part of background sampling task (ICF KE 1992).	Bioassay reference samples: Two surface water samples will be taken at a location defined by the background task (ICF KE 1992).	Not applicable

3-trichlorophenyl]urea Temp.-Temperature TOC-Total organic carbon USCS-United Soil Classification System

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TABLE 4-6 DATA QUALITY OBJECTIVES FOR CLUSTER 1 STUDY AREA

Data Quality Objective	Site Survey	Freshwater Benthic Survival	Abiotic Sampling-Sediments
Objective	A qualitative site survey will be performed to further characterize the aquatic and terrestrial habitats of Cluster 1 and the wildlife associated with these habitats.	Sediment will be collected and tested to evaluate its chronic toxicity to benthic freshwater benthic organisms. Hyallela Azteca were selected for testing because they maintain intimate contact with the sediment.	Sediment data will be collected in Monks Creek to determine the extent to which past disposal practices and maintenance operations have impacted sediment quality and could cause toxicological effects on benthic species.
Target media/group	Aquatic and terrestrial organisms	Sediment invertebrates	Sediment
Bioassay endpoint	Not applicable	Survival and growth at 10-14 days	Not applicable
Chemical data	Not applicable	Not applicable	Samples will be analyzed for TAL/TCL compounds, CSM breakdown products, explosives.
Physical data	Vegetative survey will include taxanomic identification and mapping.	Not applicable	pH will be recorded prior to sampling. Map coordinates for the sampling locations will be generated. USCS, grain size, TOC, %moisture
Sampling method	Environmental biased, grab, and intrusive.	Environmental biased, grab, and intrusive	Environmental biased, grab, and intrusive
Data use	Risk assessment	Risk assessment	Risk assessment
Data level	Physical data: Level I	Biological data: Level II	Chemical data: Level III
Chemical analytical method	Not applicable	Chemical data collected in support of bioassay is described in the Abiotic Sampling column of this table.	USEPA CLP, USAEC, USEPA SW-846
Typical detection limit	None	None	1 ppb
Quality control samples	None	Control group survival must be ≥ 90%. All reference assays must fall within control limits. Three replicates per concentration.	Trip blanks, rinse blanks, duplicates
Background samples	Not applicable	2 upgradient reference samples	2 upgradient reference samples

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TABLE 4-7 DATA QUALITY OBJECTIVES FOR MICHAELSVILLE LANDFILL STUDY AREA

DQO	Site Survey	Surface Water Investigation
Objective	A qualitative site survey will be performed to characterize principal habitat types and receptor species. Site survey will include field taxonomic identification of vegetation.	Chronic bioassays will be performed on surface water samples to evaluate potential impacts of amphibians-Rana pipiens (Birge et al., 1985).
Bioassay endpoint	Not applicable	Impacts on the amphibian population will be indicated by a statistically significant reduction in survival or increase in teratogenic effects in organisms exposed to creek water relative to reference water.
Chemical data	Not applicable	TAL/TCL, explosives, water quality
Physical data	Not applicable	Field data: Temperature, pH, salinity, dissolved oxygen, and conductivity
Sampling method	Not applicable	Environmental, biased, grab, and intrusive
Data use	Risk assessment	Risk assessment
Data level	Not applicable	Bioassay: Level II Chemical: Level III Field: Level I
Chemical analytical method	Not applicable	USEPA CLP, USATHAMA, Standard Methods
Typical detection limit	Not applicable	Water: 1µg/L
Quality control	Not applicable	Rinse blanks, duplicates, and control samples.
Background samples	During this effort, similar habitats will be examined as part of a background sampling task (ICF KE 1992).	Background sampling task will be used to obtain appropriate biotic background samples (ICF KE 1992).

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TABLE 4-8 DATA QUALITY OBJECTIVES FOR BUSH RIVER STUDY AREA

Data Quality Objective	Site Survey
Objective	Because little information is currently available for the Bush River Study Area, a survey will be conducted to identify habitat types and potential receptor species in the study area.
Chemical data	None
Physical data	A habitat map will be generated through use of aerial photographs and ground truthing. Physical descriptions of soils will be noted. In aquatic environments pH, dissolved oxygen, conductivity and/or salinity, and sediment description will be noted.
Sampling method	Environmental, biased, grab, intrusive
Data Use	Risk assessment
Data level	Physical data: Level I
Chemical analytical method	Not applicable
Typical detection limit	Not applicable
Quality control samples	None
Background samples	During this effort, similar habitats will be examined as part of a background sampling task (ICF KE 1992).

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Data Quality Objective	Site Survey
Objective	The survey will be conducted to identify habitat types and potential receptor species in the Lauderick Creek Study Area.
Chemical data	None
Physical data	A habitat map will be generated through use of aerial photographs and ground truthing. Physical descriptions of soils will be noted. In aquatic environments pH, dissolved oxygen, conductivity and/or salinity, and sediment description will be noted.
Sampling method	Environmental, biased, grab, intrusive
Data use	Risk assessment
Data level	Physical data: Level I
Chemical analytical method	Not applicable
Typical detection limit	Not applicable
Quality control samples	None
Background samples	During this effort, similar habitats will be examined as part of a background sampling task (ICF KE 1992).

TABLE 4-10 DATA QUALITY OBJECTIVES FOR OTH

DQO	Old Dump on Swan Creek	Old Dump on Woodrest Creek	Wastewater Ditch at Shell Washout Facility	DRMO Scrap Mel
Objective	A site survey will be performed to characterize habitats and potential receptors.	A site survey will be performed to characterize habitats and potential receptors.	A site survey will be performed to characterize habitats and potential receptors.	A site survey will be pe characterize habitats a receptors.
	Chronic bloassays will be performed on sediment samples to evaluate potential impact on benthic invertebrates (Hyalella azteca).	Chronic bloassays will be performed on sediment samples to evaluate potential impact on benthic invertebrates (Hyalella azteca).	Chronic bloassays will be performed on sediment samples to evaluate potential impact on benthic invertebrates (Hyalella azteca).	Chronic bioassays will performed on sediment to evaluate potential in benthic invertebrates (/ azteca).
·	Supplement analytes for RI/FS sediment chemical analytical data are proposed for WES to support risk assessment. Physical testing of sediment is proposed for identification of benthic habitats and chemical fate and transport properties.	Physical testing of sediment is proposed for identification of benthic habitats and chemical fate and transport properties.	Supplement analytes for RI/FS abiotic chemical analytical data are proposed for WES to support risk assessment. Physical testing of sediment is proposed for identification of benthic habitat and chemical fate and transport properties.	Supplement analytes for abiotic chemical analyt proposed for WES to sinisk assessment. Physiof sediment is propose identification of benthic and chemical fate and properties.
Bioassay assessment endpoints	Impacts on aquatic and benthic invertebrates will be indicated by a statistically significant reduction in survival, reproduction, or fecundity in organisms exposed to creek surface water and sediment relative to reference water.	Impacts on aquatic and benthic invertebrates will be indicated by a statistically significant reduction in survival, reproduction, or fecundity in organisms exposed to creek surface water and sediment relative to reference water.	impacts on the amphibian population will be indicated by a statistically significant reduction in survival or increase in teratogenic effects in organisms exposed to basin water relative to reference water.	Impacts on the amphib population will be indic statistically significant i survival or increase in t effects in organisms ex creek water relative to water.
Chemical data	Surface water abiotic data: WES: TAL/TCL, explosives, water quality Reference (ICF KE): TAL/TCL and water quality Sediment abiotic data: WES: RI/FS-TAL/TCL (pesticides/PCBs only), oil and grease Proposed additional data for WES: TCL VOCs, TCL BNAs, and explosives Reference (ICF KE): TAL/TCL	Surface water abiotic data: WES: TAL/TCL, explosives, water quality Reference (ICF KE): TAL/TCL and water quality Sediment abiotic data: WES: TAL/TCL, explosives Reference (ICF KE): TAL/TCL	Surface water abiotic data: WES: TAL, explosives, water quality Proposed additional data for WES: TCL Reference (ICF KE): TAL/TCL and water quality Sediment abiotic data: WES: TAL, explosives Proposed additional data for WES: TCL Reference (ICF KE): TAL/TCL	Surface water abiotic d TAL/TCL (VOCs only), c grease, TPH, water qua Proposed additional da WES: TCL BNAs, TCL pesticides/PCBs, and e Reference (ICF KE): TA water quality Sediment abiotic data: WES: TAL/TCL (VOCs c and grease, TPH Proposed additional da WES: TCL BNAs, TCL pesticides/PCBs, and e Reference (ICF KE): TA

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TABLE 4-10 VES FOR OTHER ABERDEEN AREAS

DRMO Scrap Metal Yard	Old Dump on Spesutie island	Chemical Dump Ponds on Spesulie Island	Landfill at Churchville Test Course	Harvest Survey of Aberdeen Area
ite survey will be performed to aracterize habitats and potential eptors. ronic bioassays will be formed on sediment samples evaluate potential impact on nthic invertebrates (Hyalella aca). pplement analytes for RI/FS lotic chemical analytical data is sposed for WES to support the c assessment. Physical testing sediment is proposed for intification of benthic habitats d chemical fate and transport sperties.	A site survey will be performed to characterize habitats and potential receptors. Physical testing of sediment is proposed for identification of benthic habitats and chemical fate and transport properties.	A site survey will be performed to characterize habitats and potential receptors. Physical testing of sediment is proposed for identification of benthic habitats and chemical fate and transport properties.	A site survey will be performed to characterize habitats and potential receptors. Physical testing of sediment is proposed for identification of benthic habitats and chemical fate and transport properties.	Tissue residue analyses will be performed on muscle, liver, and fur of deer (10) and muskrat (10) to determine extent of uptake of chemicals originating from Aberdeen Area. Data from deer muscle will be used to evaluate human exposure. Histological analyses will be performed on liver and muscle of deer (3) and muskrat (3) to determine if impacts are occurring to the Individual organisms, and if those impacts are likely to affect the viability of the population.
pacts on the amphibian pulation will be indicated by a distically significant reduction in rival or increase in teratogenic ects in organisms exposed to sek water relative to reference ter.	Not applicable	Not applicable	Not applicable	Not applicable
rface water abiotic data: WES: L/TCL (VOCs only), oil and ease, TPH, water quality pposed additional data for ES: TCL BNAs, TCL sticides/PCBs, and explosives ference (ICF KE): TAL/TCL and atter quality diment abiotic data: ES: TAL/TCL (VOCs only), oil d grease, TPH oposed additional data for ES: TCL BNAs, TCL sticides/PCBs, and explosives ference (ICF KE): TAL/TCL	Surface water ablotic data: WES: TAL/TCL, explosives, water quality Sediment ablotic data: WES: TAL/TCL, explosives	Surface water abiotic data: WES: TAL/TCL, explosives, water quality Sediment abiotic data: WES: TAL/TCL, explosives	Surface water abiotic data: WES: TAL/TCL, explosives, water quality Sediment abiotic data: WES: TAL/TCL, explosives	ICF KE: Muscle and liver will be analyzed for TAL, explosives, TCL pesticides/PCBs, and lipid content. ICF KE: Fur samples will be analyzed for mercury and TCL pesticides.

TABLE 4 DATA QUALITY OBJECTIVE

Dao	Old Dump on Swan Creek	Old Dump on Woodrest Creek	Wastewater Ditch at Shell Washout Facility	D
Physical data	Data collected during site survey (i.e., water characteristics (salinity, DO, pH, conductivity))	Data collected during site survey (i.e., water characteristics (salinity, DO, pH, conductivity))	Data collected during site survey (i.e., water characteristics (salinity, DO, pH, conductivity))	Data c (i.e., v DO, pi
	Map coordinates will be generated by RI/FS contractor.	Map coordinates will be generated by RI/FS contractor.	Map coordinates will be generated by RI/FS contractor.	Map c genen
·	Physical testing: USCS, grain size, TOC, % solids	Physical testing: USCS, grain size, TOC, % solids	Physical testing: USCS, grain size, TOC, % solids	Physic size, T
Sampling method	Environmental, biased, grab,	Environmental, biased, grab, intrusive	Environmental, biased, grab, Intrusive	Enviro intrusi
Data use	Risk assessment	Risk assessment	Risk assessment	Risk a
Data level	Bioassay: Level II Chemical: Level III Physical: Level I	Bioassay: Level II Chemical: Level III Physical: Level I	Bioassay: Level II Chemical: Level III Physical: Level I	Bioass Chemi Physic
Chemical analytical method	USEPA CLP, Standard Methods	USEPA CLP, Standard Methods	USEPA CLP, Standard Methods	USEP
Typical detection	Water: 1 g/L Sediment: 1 mg/kg	Water: 1 g/L Sediment: 1 mg/kg	Water: 1 g/L Sediment: 1 mg/kg	Water Sedim
Quality control samples	Trip blanks, rinse blanks, and duplicates (WES 1991)	Trip blanks, rinse blanks, and duplicates (WES 1891)	Trip blanks, rinse blanks, and duplicates (WES 1991)	Trip b
	Controls for bioassays (ICF KE)	Controls for bioassays (ICF KE)	Controls for bloassays (ICF KE)	Contro
Background samples	One sample will be collected as the control sample for bioassays.	One sample will be collected as the control sample for bioassays.	One sample will be collected as the control sample for bloassays.	One s the co
	Background sampling task will be used to obtain appropriate abiotic background samples (ICF KE 1992a,b).	Background sampling task will be used to obtain appropriate abiotic background samples (ICF KE 1992a,b).	Background sampling task will be used to obtain appropriate abiotic background samples (ICF KE 1992a,b).	Backg used 1 backg 1992a

BNAs Base neutral acids
TPH Total petroleum hydrocarbons

DO Dissolved oxygen USCS United Soll Classification System ICF KE ICF Kaiser Engineers, Inc. VOCs Volatile organic compounds

PCBs Polychlorinated TOC Total organic ca

BLE 4-10 (continued) CTIVES FOR OTHER ABERDEEN AREAS

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DRMO Scrap Metal Yard	Old Dump on Spesutie Island	Chemical Dump Ponds on Spesutie Island	Lendfill at Churchville Test Course	Harvest Survey of Aberdeen Area
Data collected during site survey (i.e., water characteristics (salinity, DO, pH, conductivity)) Map coordinates will be generated by Ri/FS contractor. Physical testing: USCS, grain size, TOC, % solids	Data collected during site survey (i.e., water characteristics (salinity, DO, pH, conductivity) Map coordinates will be generated by RI/FS contractor. Physical testing: USCS,	Data collected during site survey (i.e., water characteristics (salinity, DO, pH, conductivity)) Map coordinates will be generated by RI/FS contractor. Physical testing: USCS,	Data collected during site survey (i.e., water characteristics (salinity, DO, pH, conductivity)) Map coordinates will be generated by RI/FS contractor. Physical testing: USCS,	Anomalies to reproductive structures and other internal organ lesions, in addition to skeletal abnormalities
Environmental, biased, grab, intrusive	grain size, TOC, % solids Environmental, biased, grab, intrusive	grain size, TOC, % solids Environmental, blased, grab, intrusive	grain size, TOC, % solids Environmental, blased, grab, intrusive	Environmental, blased, grab, intrusive
Risk assessment	Risk assessment	Risk assessment	Risk assessment	Risk assessment
Bioassay: Level II Chemical: Level III Physical: Level I	Bioassay: Level II Chemical: Level III Physical: Level I	Bioassay: Level II Chemical: Level III Physical: Level I	Bioassay: Level II Chemical: Level III Physical: Level I	Chemical: Level IV Physical: Level I
USEPA CLP, Standard Methods	Not applicable	Not applicable	Not applicable	USEPA 1986b, Hausknecht et al., USAEC, ASTM
Water: 1 g/L Sediment: 1 mg/kg	Water: 1 g/L Sediment: 1 mg/kg	Water: 1 g/L Sediment: 1 mg/kg	Water: 1 g/L Sediment: 1 mg/kg	Tissue: 1 mg/kg
Trip blanks, rinse blanks, and duplicates (WES 1991) Controls for bloassays (ICF KE)	Trip blanks, rinse blanks, and duplicates (WES 1991)	Trip blanks, rinse blanks, and duplicates (WES 1991)	Trip blanks, rinse blanks, and duplicates (WES 1991)	None
One sample will be collected as the control sample for bloassays. Background sampling task will be used to obtain appropriate abiotic background samples (ICF KE 1992a,b).	Background sampling task will be used to obtain appropriate abiotic background samples (ICF KE 1992a,b).	Background sampling task will be used to obtain appropriate abiotic background samples (ICF KE 1992a,b).	Background sampling task will be used to obtain appropriate ablotic background samples (ICF KE 1992a,b).	Background sampling task will be used to obtain appropriate biotic background samples (ICF KE 1992a,b).

ilorinated biphenyls iganic carbon

TAL/TCL USEPA Target Analyte List/Target Compound List WES U.S. Army Corps of Engineers Waterways Experiment Station



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TABLE 4-11 DATA QUALITY OBJECTIVES FOR GRACES QUARTERS STUDY AREA

Data Quality Objective	Site Survey
Objective	The survey will be conducted to identify habitat types and potential receptor species in the Graces Quarters study area.
Bioassay endpoint	Not applicable
Chemical data	None
Physical data	A habitat map will be generated through use of aerial photographs and ground truthing. Physical descriptions of soils will be noted. In aquatic environments pH, dissolved oxygen, conductivity and/or salinity, and sediment description will be noted.
Sampling method	Environmental, biased, grab, intrusive
Data use	Risk assessment
Data level	Physical data : Level I
Analytical method	Not applicable
Typical detection limit	Not applicable
Quality control samples	None
Background samples	During this effort, similar habitats will be examined as part of a background sampling task (ICF KE 1992a).

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DQO	Site Survey	Surface Water investigation
Objective	A qualitative site survey will be performed to characterize principal habitat types and receptor species. Site survey will include field taxonomic identification of vegetation.	Five (5) surface water samples will be collected in order to determine if contamination is present at one surface water drainage area not addressed by the RI/FS.
Chemical data	Not applicable	TAL/TCL, explosives, water quality
Physical data	Not applicable	Field data: Temperature, pH, salinity, dissolved oxygen, and conductivity
Sampling method	Not applicable	Environmental, grab
Data use	Risk assessment	Risk assessment
Data level	Not applicable	Chemical: Level III Field: Level I
Chemical analytical method	Not applicable	USEPA CLP, USATHAMA, Standard Methods
Typical detection limit	Not applicable	Water: 1µg/L
Quality control	Not applicable	Duplicates
Background samples	During this effort, similar habitats will be examined as part of a background sampling task (ICF KE 1992).	Background sampling task will be used to obtain appropriate biotic background samples (ICF KE 1992).

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TABLE 4-13 DATA QUALITY OBJECTIVES FOR OTHER CREEKS STUDY AREA

DQO	Site Survey	Sediment investigation
Objective	A qualitative site survey will be performed to characterize principal habitat types and receptor species. Site survey will include field taxonomic identification of vegetation.	Chronic bioassays using Hyalella azteca will be performed on sediment samples collected from Woodrest Creek, Swan Creek. Data will be used to determine if sediment is impacted by suface run-off from the surrounding watershed. To support bioassays, samples will be collected for chemical analysis. In addition, samples for chemical analysis to determine if contamination is present will be collected from Dipper Creek, Mosquito Creek Spesutie Narrows, and Delph Creek.
Bioassay endpoint	Not applicable	Impacts on the amphipods will be indicated by a statistically significant reduction in survival or increase in teratogenic effects in organisms exposed to creek water relative to reference water.
Chemical data	Not applicable	TAL/TCL, explosives, water quality
Physical data	Not applicable	Field data: Temperature, pH, salinity, dissolved oxygen, and conductivity
Sampling method	Not applicable	Environmental, grab
Data use	Risk assessment	Risk assessment
Data level	Not applicable	Bioassay: Level II Chemical: Level III Field: Level I
Chemical analytical method	Not applicable	USEPA CLP, USATHAMA, Standard Methods
Typical detection limit	Not applicable	Water: 1μg/L
Quality control	Not applicable	Rinse blanks, duplicates.
Background samples	During this effort, similar habitats will be examined as part of a background sampling task (ICF KE 1992).	Background sampling task will be used to obtain appropriate biotic background samples (ICF KE 1992).

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TABLE 4-14 DATA QUALITY OBJECTIVES FOR THE OTHER EDGEWOOD STUDY AREAS

Data Quality Objective	Site Survey
Objective	Because little information is currently available for the Other Edgewood Study Area, a survey will be conducted to identify habitat types and potential receptor species in the study area.
Chemical data	None
Physical data	A habitat map will be generated through use of aerial photographs and ground truthing. Physical descriptions of solls will be noted. In aquatic environments pH, dissolved oxygen, conductivity and/or salinity, and sediment description will be noted.
Sampling method	Environmental, biased, grab, intrusive
Data Use	Risk assessment
Data level	Physical data: Level I
Chemical analytical method	Not applicable
Typical detection limit	Not applicable
Quality control samples	None
Background samples	During this effort, similar habitats will be examined as part of a background sampling task (ICF KE 1992).

SECTION 5.0

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5.0 SAMPLING

Sample labeling, container requirements, preservation, sample collection, sample custody, and field calibration are described in this section. Procedures described are designed to eliminate external contamination and to ensure data quality through the use of SOPs. References to methods of collection and detailed SOPs are provided in Appendix A.

5.1 SAMPLE LABELING

Each aqueous and solid sample will be assigned a unique sequential number at the time of sampling, which will be permanently affixed to the sample container. The sample label will include the following information:

- Sample number;
- Sampling date;
- Preservative, if applicable;
- Analyte(s);
- Sampler's initials; and
- Installation name.

Labels will be covered with polyethylene tape to prevent the loss of the label during shipment. The SOP provided in Appendix A details procedures for completing sample labels.

5.2 CONTAINERS

All sample containers will be cleaned prior to use by the contract laboratory in accordance with USEPA protocols as described in Section 6.1.1. The sample containers to be used for the various analyses for the APG TERA are provided in tabular format for chemical analyses of aqueous samples (Table 5-1), chemical analysis of soil and sediment samples (Table 5-2), chemical analysis of tissue samples (Table 5-3), samples for physical testing (Table 5-4), aqueous samples for bioassays (Table 5-5), and soil and sediment samples for bioassays (Table 5-6). All container requirements meet method/laboratory specifications.

5.3 SAMPLE PRESERVATION

Preservatives will be required to retard hydrolysis of chemical compounds and complexes, to reduce volatility of constituents, and to retard biological action during transit and storage prior to laboratory analysis. Preservatives will be added to appropriate samples at the time of collection. The types of preservation required for samples collected during this project are contained in Tables 5-1 (chemical aqueous), Table 5-2 (chemical solid), Table 5-3 (tissue samples), Table 5-4 (physical analysis), Table 5-5 (bioassay aqueous), and Table 5-6 (bioassay solid). In addition to chemical preservatives, all samples for chemical analysis will be transported to the laboratory temperature controlled coolers. Ice will be used to maintain the internal cooler temperature required for preservation. Procedures for chemical sample preservation are described below:

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TABLE 5-1 SAMPLE PRESERVATION, BOTTLE REQUIREMENTS, AND HOLDING TIMES FOR AQUEOUS SAMPLES

	Analyte		Required Headspace	Preservative	Holding Time
	VOCs	40 mL VOA vial	0%	Cool to 4°C	7 days ¹
		· in duplicate	i	Cool to 4°C pH<2, HCl ¹	14 days
TCL	Semivolatiles	1-L glass, with teflon lined cap	10%	Cool to 4°C Protect from light	7 days to extraction, 40 days to analysis
	Pesticides/PCBs	1-L glass, with teflon lined cap	10%	Cool to 4°C Protect from light	7 days to extraction, 40 days to analysis
TAL	Metals	1-L glass or	10%	pH<2, HNO ₃	180 days
	Mercury	polyethylene			28 days
	Cyanide	1-L glass or polyethylene	10%	pH>12, NaOH Cool to 4°C	12 days
CSM deg	radation products	3-1L amber glass	10%	Cool to 4°C	7 days
	Chloride	1-L polyethylene	10%	Cool to 4°C	28 days
	sulfate	1-L polyethylene	10%	Cool to 4°C	28 days
	nitrate/nitrite	1-L polyethylene	10%	pH<2,H ₂ SO ₄ Cool to 4°C	28 days
	Total phosphorus	1-L polyethylene	10%	pH<2, H ₂ SO ₄ Cool to 4°C	28 days
Water quality	Alkalinity	1-L polyethylene	10%	Cool to 4°C	14 days
y	Hardness	1-L polyethylene	10%	pH<2, HNO ₃ or H ₂ SO ₄ Cool to 4°C	6 months
	Total suspended solids	1-L polyethylene	10%	Cool to 4°C	48 hours
	Biological oxygen demand	1-L polyethylene	10%	Cool to 4°C	48 hours

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TABLE 5-1 (continued) SAMPLE PRESERVATION, BOTTLE REQUIREMENTS, AND HOLDING TIMES FOR AQUEOUS SAMPLES

	Analyte	Bottle Requirement and Volume	Required Headspace	Preservative	Holding Time
Water	Chemical oxygen demand	1-L polyethylene	10%	pH<2, H ₂ SO ₄ Cool to 4°C	28 days
quality (cont.)	Ammonia	1-L polyethylene	10%	pH<2, H ₂ SO ₄ Cool to 4°C	28 days
	Explosives	2-1L amber glass	10%	none	7 days to extraction; 40 days to analysis

CSM Chemical surety materials
PCBs Polychlorinated biphenyls
TAL Target analyte list
TCL Target compound list
VOCs Volatile organic compounds

In areas where the presence of Mustard Agent is suspected to be present (Study areas located with in the Edgewood Area of APG), HCl will not be added to VOCs as a preservative.

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TABLE 5-2 SAMPLE PRESERVATION, BOTTLE REQUIREMENTS, AND HOLDING TIMES FOR SEDIMENT/SOIL SAMPLES

Analyte		Bottle Requirement and Volume	Required Headspace	Preservative	Holding Time
	VOCs	4 oz, glass with teflon cap	0%	Cool to 4°C, protect from light	14 days to extraction
TCL	Semivolatiles	4 oz, glass with teflon cap	10%	Cool to 4°C, protect from light	14 days to extraction
	Pesticides/PCBs	4 oz, glass with teflon cap	10%	Cool to 4°C, protect from light	14 days to extraction
TAL	Metals	1-L glass or	10%	Cool to 4°C	180 days
	Hg	polyethylene			28 days
	Cyanide	1-L glass or polyethylene	10%	Cool to 4°C	12 days
CSM degradation products		3-1 L amber glass	10%	Cool to 4°C	7 days to extraction, 40 days to analysis
Explosives		2-1 L amber glass	10%	Cool to 4°C	7 days to extraction, 40 days to analysis
Dioxin/furans		2-1 L amber glass	10%	Cool to 4°C	30 days to extraction, 45 days to analysis
Organomercury		1-1 L polyethylene	10%	Cool to 4°C	28 days
TCPU		1-1 L amber glass	10%	Cool to 4°C	14 days

CSM

Chemical surety materials

PCBs

Polychlorinated biphenyls

TAL

Target analyte list
Target compound list

VOCs

Volatile organic compounds

TCPU

Trichlorophenylurea

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TABLE 5-3 SAMPLE PRESERVATION, BOTTLE REQUIREMENTS, AND HOLDING TIMES FOR TISSUE SAMPLES

Analyte	Bottle and Mass Requirement	Preservative For Shipment	Holding Time
TCL Semivolatile Organic Compounds	Aluminum Foil 30g Biomass	Freeze with dry ice	No holding times have been established by the USEPA for the analysis of tissue samples.
TCL Pesticides/PCBs	Aluminum Foil 15g Biomass	Freeze with dry ice	No holding times have been established by the USEPA for the analysis of tissue samples.
TAL Metals	Aluminum Foil 15g Biomass	Freeze with dry ice	No holding times have been established by the USEPA for the analysis of tissue samples.
Cyanide	Aluminum Foil 10g Biomass	Freeze with dry ice	No holding times have been established by the USEPA for the analysis of tissue samples.
Explosives	Aluminum Foil 10g Biomass	Freeze with dry ice, Protect from Light	No holding times have been established by the USEPA for the analysis of tissue samples.
Organosulfur Compounds	Aluminum Foil 10g Biomass	Freeze with dry ice	No holding times have been established by the USEPA for the analysis of tissue samples.
DIMP, DMMP	Aluminum Foil 10g Biomass	Freeze with dry ice	No holding times have been established by the USEPA for the analysis of tissue samples.
IMPA, MPA	Aluminum Foil 10g Biomass	Freeze with dry ice	No holding times have been established by the USEPA for the analysis of tissue samples.
Thiodiglycol	Aluminum Foil 10g Biomass	Freeze with dry ice	No holding times have been established by the USEPA for the analysis of tissue samples.
Organomercuric Compounds	Aluminum Foil 15g Biomass	Freeze with dry ice	No holding times have been established by the USEPA for the analysis of tissue samples.

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TABLE 5-4 SAMPLE PRESERVATION, BOTTLE REQUIREMENTS, AND HOLDING TIMES FOR PHYSICAL SAMPLES

Analyte	Bottle Requirement	Required Headspace	Preservative	Holding Time
USCS	1L mason jar	10%	none	none
Grain size	1L mason jar	10%	none	none
Total organic carbon	1L mason jar	10%	none	28 days
Percent moisture	1L mason jar	0%	Bottle lid wrapped with teflon tape to seal in moisture	none

USCS United Soil Classification System

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TABLE 5-5 SAMPLE PRESERVATION, BOTTLE REQUIREMENTS^a, AND HOLDING TIMES FOR AQUEOUS BIOASSAY SAMPLES^b

Species for Chronic Study	Volume Requirement ^c	Required Headspace	Preservative	Holding Time
Mysidopsis bahia	600 mL	10%	Cool to 4°C	72 hrs
Rana pipiens	3250 mL	10%	Cool to 4°C	72 hrs
Cyprinodon variegatus	1200 mL	10%	Cool to 4°C	72 hrs
Daphnia magna	600 mL	10%	Cool to 4°C	72 hrs
Pimephales promelas	1200 mL	10%	Cool to 4°C	72 hrs
Selenastrum capricornutum	600 mL	10%	Cool to 4°C	72 hrs

All samples will be collected in polyethylene cubitainers.

b USEPA 1989

The above values are the volume requirements for bioassay study. Additional volumes will be required and specified by the laboratory for laboratory quality control studies.

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TABLE 5-6 SAMPLE PRESERVATION, BOTTLE REQUIREMENTS^a, AND HOLDING TIMES FOR SEDIMENT BIOASSAY SAMPLES

Species for Chronic Study	Volume Requirement ^b	Required Headspace	Preservative	Holding Time
Hyalella azetec	300g	10%	Cool to 4°C	72 hrs
Chironomus tentans	300 g	10%	Cool to 4°C	72 hrs

^a All samples will be collected in polyethylene cubitainers.

The above values are the approximate volume requirements for bioassay studies. Additional volumes will be required and specified by the laboratory for laboratory quality control studies.

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- 1. Preservatives will be added to samples either using a pipette or directly to the sample if vials of preservatives are used.
- 2. The sample bottle will be capped, and the bottle gently agitated in order to homogenize the preservative throughout the sample.
- 3. The sample bottle cap will be reopened and a small amount of the sample will be transferred to a beaker and the bottle will be closed.
- 4. Either pH paper or an electronic pH meter will be used to determine the pH of the sample, where applicable. pH paper or a pH meter will never be put directly into the sample bottle in order to avoid contamination from entering or leaving the sample.
- 5. If the proper pH has been reached (where applicable), the sample bottle will remain closed. If the proper pH has not been reached, the sample bottle will be reopened, more preservative added, the bottle shaken, and the pH tested until the proper pH has been reached.

For VOCs the above steps will be performed on a dummy sample in order to determine the amount of preservative necessary to reach the desired pH. The same amount will be added to the vials before the VOC fraction is collected. Once the VOC fraction is in the vial, the vial will be sealed with 0% headspace.

5.4 SAMPLE COLLECTION

Detailed procedures for the collection of samples for chemical analysis are provided as SOPs in Appendix A, and are summarized in the Technical Plan for Risk and Biological Impact Assessment (ICF KE 1992), thus they will not be discussed further in this document. Collection of all samples will follow standard USEPA CLP and USAEC protocols. This section discusses the collection of quality control samples.

5.4.1 Quality Control Samples Collected in the Field

Field operations performed during the APG TERA will include the collection of several types of quality control samples. These samples will include duplicates, rinse blanks/equipment blanks, and trip blanks.

Duplicate samples will be taken from areas which are known or suspected to be contaminated and will be collected at a frequency of 10% of all field samples. Fractions for the same analytical parameters will always be collected consecutively.

Rinse blanks will be collected when the sampling equipment is decontaminated and reused in the field or when a sample collection vessel (bailer or beaker) will be used. A consistent volume of demonstrated analyte free water (High Performance Liquid Chromatography (HPLC)-grade for organics, deionized for inorganics) will be poured over the equipment (i.e., rinsing the equipment) collecting the water in a sample container. The rinse blank determines whether the decontamination procedure has

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been adequately performed and that there is no cross-contamination of samples occurring due to the equipment itself. Analysis of rinse blanks will be for all analytes of interest. Rinse blanks will be collected at a frequency of 5% of the total number of samples.

Trip blanks will be provided by the contract laboratory and will consist of demonstrated analyte free HPLC-grade water sealed in 40 mL teflon-lined septum vials. The trip blank is used to determine if any on-site atmospheric contaminants are seeping into the sample vials, or if any cross-contamination of samples is occurring during shipment or storage of sample containers. The trip blanks will accompany the aqueous samples for VOC analysis to the laboratory.

5.5 SAMPLE DOCUMENTATION

5.5.1 Field Parameter Forms

Upon collection of a sample for chemical analysis a field parameter form (FPF), must be completed. All forms must be filled out using a ball point pen. The FPF must be signed by the field member(s) responsible for the sample collected. The FPF will accompany the sample to the laboratory and will contain the following information:

- a. Installation/Site and Area;
- b. Installation Code;
- c. File name:
- d. Site type;
- e. Site ID;
- f. Field sample number;
- g. Date/Time (military format);
- h. Sample Program;
- i. Depth of sample collected/Depth interval/Units;
- j. Sample Method (IRDMIS Coded);
- k. Field measurements and calibration reference;
- I. Requested Analytes;
- m. Sample Container/Number of containers;
- n. Laboratory ID; and

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o. Preservation information.

A sample FPF is presented in Figure 5-1.

5.5.2 Chain-of-Custody

Chain-of-Custody (COC) forms will accompany sample containers in the field, during transit to the laboratory, and upon receipt by the laboratory. The SOP provided in Appendix A describes the procedure for completing the form.

5.5.3 Field Logbook

All information pertinent to the sampling effort will be recorded in a bound logbook or equivalent standardized form. Each page/form will be consecutively numbered. All entries will be made in indelible ink and all corrections will consist of line-out deletions that are initialed and dated. At a minimum, entries in the field logbook will include the following:

- a. Date and time of field activity;
- b. Field event (i.e. decontamination of equipment, sampling event);
- c. Identification of sample crew members;
- d. Location and description of each sampling point;
- e. Sample type (i.e. groundwater, surface water)
- f. Details of the sample site (for example unusual odors, weather conditions, etc);
- g. Unique sequential field sample number;
- h. Requested analytes;
- i. Sample technique (for example grab or composite);
- j. Sample preservation
- I. Any field measurements made.

5.6 SAMPLE TRANSPORTATION

Evidence of sample custody shall be traceable from the time the cleaned sample containers leave the laboratory until filled sample containers are transmitted back to the laboratory. To achieve this condition, custody seals and chain-of-custody documentation will accompany all sample containers.

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FIELD PARAMETER/LOGBOOK FORM SOIL AND SEDIMENT SAMPLES

						, , , , , , , , , , , , , , , , , , ,
HIGH C	ONCENTRATION EX	CPECTED?			ID?	
INSTALL	ATION/SITE			AREA		
		FILE NAME SITE TYPE				
SITE ID		FIE	LD SAMPL	E NUMBER		
DATE (N	IM/DD/YY)	_// TI	ME	AM PM	SAMPL PROG.	
DEPTH ((TOP)	DEPTH IN	TERVAL _		UNITS	
		SAM	PLE METH	IOD		
SPLIT SP	OON AUC	GER SHELBY TU		_ SCOOP	OTHER	
СНК	ANALYSIS				REMARKS	
						
	***************************************			•		

			TOTAL N	UMBER OF CONTAI	INERS FOR SAMPLE	
TEATHER	/TEMPERATURE			SAME	PLER	

FIGURE 5-1
SAMPLE FIELD PARAMETER FORM

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5.6.1 Sample Documents

After the sample containers are filled, the COC form will be completed in triplicate (carbonless copies), checked against the contents of the cooler, and the original (white) and the yellow copy will be placed in a plastic bag, and taped inside the secured container to be shipped to the laboratory. The pink copy of the COC form will be retained by the Task Manager. The original COC form will be transmitted to the Project Data Coordinator (PDC) by the laboratory after samples have been analyzed.

5.6.2 Custody Seals

Custody seals will be signed and dated and affixed to shipment containers containing sample bottles from the laboratory. After the cooler has been properly secured, custody seals will be placed across the cooler opening to ensure the integrity of the samples during shipment.

5.6.3 Sample Receipt

Samples delivered to the lab will be accepted by the laboratory technician. Samples can be accepted Monday through Friday. Special arrangements will be required if Saturday delivery is necessitated. Chain-of-Custody for laboratory receipt will be established in the following manner:

- 1. The carrier and the time of arrival is documented in the daily receipt log. The number of items on the COC is checked with the actual number received to ensure that all samples arrived.
- 2. Notation is made as to whether the shipping container (cooler) was sealed with custody seals.
- 3. The cooler is opened, the internal ambient temperature of the cooler taken, and the samples are itemized. All deviations are noted and reported to the laboratory QA Coordinator.
- 4. Lot numbers will be assigned to the samples. Reference to field numbers will be documented in the appropriate logbook. All data are entered into the computer tracking system, with analyses required by holding-time specified dates.

5.6.4 <u>Laboratory Receipt</u>

Once the sample has been transmitted to the laboratory the following sequence of events will occur:

- 1. The samples are recorded on the Sample Log-In Form to summarize all the information pertaining to the sample/order to instruct the laboratory on the proper analysis and reporting of samples.
- 2. After the samples are logged in, they are assigned to the appropriate storage refrigerator.

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- 3. All transfers of samples into and out of storage are documented.
- 4. Samples remain in secured storage until removed for sample preparation or analysis.
- 5. A refrigeration log must be generated to ensure refrigerators/freezers are operating at the appropriate temperature. The log must indicate the ambient internal temperature as well as the initials of the person recording the reading and the date. Should the temperature fluctuate outside of the specified holding time temperature range, corrective action must be taken immediately.

5.7 FIELD EQUIPMENT CALIBRATION

The proper calibration and documentation of field equipment is designed to assure that the field equipment is functioning optimally. Equipment logbooks are required to record usage, maintenance, calibration, and repair.

5.7.1 Frequency of Field Calibration

Field instrumentation/equipment will be calibrated in the field by the following schedule:

- **5.7.1.1 Photoionization Detectors and Organic Vapor Analyzers.** Calibrated upon arrival to the site and daily while in the field. Measurements of background VOCs will be documented and zeroed out, the calibration gas will be added, the reading documented, and the instrument will be adjusted for proper calibration. The final reading will also be documented. Calibration protocols and measurement will be documented in a bound logbook that accompanies each instrument.
- **5.7.1.2 Conductivity and pH Meters.** Calibrated upon arrival and departure of the site, daily while in the field. Meter will be calibrated more frequently if temperature changes by 5°C or more. The calibration of pH and conductivity meters will include an initial measurement prior to calibration each day, a measurement after calibration, and measurement at the end of the day. All measurements will be documented at the end of the field parameter form logbook or in separate calibration log forms.
- **5.7.1.3 Hydrolab.** All parameters will be calibrated each day upon arrival to the site. The parameters include dissolved oxygen, pH, temperature, and conductivity. All measurements will be documented at the end of the field parameter form logbook or in separate calibration log forms.

5.7.2 Calibration Standards

Equipment will be calibrated with the appropriate standards specified below, to be provided to the field team by the ICF KE Equipment Manager. Analytical accuracy is traceable to Standard Analytical Reference Materials (SARMs) from the National Bureau of Standards (NBS).

• Conductivity Solution: 1,000 Micromho/CM (+/- 0.50%) at 25.00°C, .053% Potassium Chloride, .0002% Iodine, and Water (CAS 7732-18-5).

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pH Buffers:

4.00 +/- 0.01 @ 25°C, Color coded red. Potassium Hydrogen Phthalate (CAS 877-24-7), Formaldehyde (CAS 50-00-0), Water (CAS 7732-18-5).

7.00 +/- 0.01 @ 25°C, Color coded yellow. Sodium Phosphate, Dibasic (CAS 7558-79-4), Potassium Phosphate, Monobasic (CAS 7778-77-0), Water (CAS 7732-18-5).

10.00 +/- 0.02 @ 25°C, Color coded blue. Potassium Borate, Tetra (CAS 1332-77-0), Potassium Carbonate (CAS 584-08-7), Potassium Hydroxide (CAS 1310-58-3), Sodium (di) Ethylenediamine Tetraacetate (CAS 6381-92-6), Water (CAS 7732-18-5).

Photoionization Detector Standards:
 Isobutylene (I-C₄H₈) 100 ppm +/- 5%, balance: Air.

SECTION 6.0

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6.0 SAMPLE ANALYSIS

In this section, sample management, holding times, preparation, instrument calibration, analytical procedures, and data management procedures are discussed.

6.1 SAMPLE MANAGEMENT

The contract laboratory will provide the following for field sampling:

- a. Cleaned sample containers:
- b. Shipping containers with custody seals;
- c. Sample preservatives;
- d. Sample labels; and
- e. Trip blanks.

6.1.1 Sample Container Cleaning

The integrity of containers for samples collected as part of the TERA is ensured by using the appropriate cleaning techniques. The contract laboratory will clean the sample bottles according to the approved methodology.

6.1.2 Shipping Containers and Custody Seals

Cleaned sample bottles will be sent to the field in shipping containers which will be used for return of samples to the laboratory. When returning full sample bottles to the laboratory, each cooler will contain packing material and sufficient ice to ensure that internal ambient temperature of 4°C are maintained for transport of the samples to the laboratory. Sample coolers will be sealed with chain-of-custody seals. In addition each sample container will have an associated chain-of-custody form (COC) and logbook entry.

6.1.3 Sample Preservatives

Preservatives will be included in the shipping container sent to the field. Unused preservative will be sent back to the laboratory when sampling has been completed in Department of Transportation (DOT)-approved containers.

6.2 SAMPLE HOLDING TIMES

Sample holding time, (the time interval between sampling and analysis), in which a sample can be considered valid and representative of the sample matrix, is based on the analytes of interest. The allowable holding times are shown in Tables 5-1 through 5-6. The laboratory tracking system should be designed to ensure that holding times are not exceeded.

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6.3 SAMPLE PREPARATION

Once the samples have been received by the laboratory, the information on the sample labels will be transcribed to a bound notebook. Sample receipt conditions, analytical parameters, analysis dates, and storage locations are entered into the existing laboratory data management system for each sample shipment. Analytical lots will be established and coding assigned in lot sequence during the logging-in stage. Samples will be securely stored at 4°C from the time of receipt through final analysis. Samples will be stored until released by the USAEC Project Officer or until the end of the contract.

Samples will be prepared, extracted, and/or spiked with reference materials or surrogate standards, as required for each specific analytical method. Percent moisture will be determined for each soil sample.

Water used in the course of organic analyses shall conform to ASTM Type II grade in accordance with guidelines specified in the USATHAMA QA Program (1990). ASTM Type I grade water will be used for inorganic analyses.

6.4 CALIBRATION

Prior to sample analysis, chemical calibration of each target analyte must be performed to ensure analytical instrumentation is functioning within the established sensitivity range. Protocols defining the procedures and QC measurements for instrument calibration should be done in accordance with criteria specified in the USATHAMA QA Program (1990).

6.4.1 initial Calibration

Initial calibrations for the methods to be used in this project are performed routinely by the laboratory as part of the USAEC certification program. Initial calibrations are not required unless the instrument fails the daily calibration procedure.

6.4.2 Daily Calibration

Prior to analysis, all instruments will be calibrated to ensure that the instrumental response has not changed from the previous calibration. Analysis should be performed on the highest concentration standard. A response within two standard deviations of the mean response for the same concentration as determined from precertification, certification, and prior initial/daily calibrations, does not warrant recalibration of the system. Should the response fail the criteria, the daily standard must be reanalyzed. Failure of the second analysis requires initial calibration to be performed as specified in the USATHAMA QA Program (1990).

6.5 SOLUTION VALIDATION

All calibration solutions and standards to be used in this program will be prepared and maintained under the normal laboratory standards tracking system. This system ensures preparation,

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checking, documentation, storage, and disposal of standards according to specified procedures and schedules appropriate for each analyte of interest.

6.6 LABORATORY ANALYTICAL PROCEDURES

USEPA CLP, USEPA SW-846, Standard Methods (SM), American Society for Testing and Materials (ASTM), and USAEC analytical methods will be used for analysis of samples, as applicable. Summary of the methods to be used as part of the TERA are provided in Table 6-1 (chemical analysis of surface water, soil, sediment and tissue), Table 6-2 (physical testing methods), and Table 6-3 (bioassay methods). This section provides brief summaries of the analytical methods as well as the analytes associated with each method. ESE Laboratory's USAEC methods are provided in Appendix D.

6.6.1 Methods for the Analysis of Aqueous and Sediment Samples

The following sections are descriptions of the methods that will be used for the analyses of aqueous and sediment samples.

6.6.1.1 Inorganic Analysis.

a. TAL Metals

TAL metals listed in Table 6-4, will be analyzed in accordance with USEPA CLP methodologies (USEPA 1991b). The metal constituents will be analyzed using one of the following methodologies: inductively coupled argon emission plasma spectroscopy (ICAP), graphite furnace atomic absorption spectroscopy (GFAA), or cold vapor atomic absorption (CVAA).

The ICAP method involves the simultaneous or sequential multi-element determination of trace elements in solution. The basis of the method is the measurement of atomic emission by optical spectrometry. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radiofrequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer and the intensities of the line are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is utilized to compensate for variable background contribution to the determination of trace elements. Background is measured adjacent to analyte lines on samples during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used will be free of spectral interference and will reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction will not be required in cases of line broadening where a background correction measurement would actually degrade the analytical result. Interferences will also be recognized and appropriate corrections made.

In order to obtain reporting limits lower than those provided by the ICAP method, arsenic, lead, and selenium will be analyzed using GFAA. GFAA involves the digestion of a representative sample

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TABLE 6-1 SUMMARY OF CHEMICAL ANALYTICAL METHODS

Analyte		Aqueous Matrix	Solid Matrix	Biological Matrix	
	VOCs	CLP SOW OLM01.0 (USEPA 1991c)	CLP OLM02.0 (USEPA 1991c)	USEPA 1986b	
TCL	Semivolatiles	. CLP SOW OLM01.0 (USEPA 1991c)	CLP SOW OLM01.0 (USEPA 1991c)	USEPA 1986b	
	Pesticides/PCBs	CLP SOW OLM01.0 (USEPA 1991c)	CLP SOW OLM01.0 (USEPA 1991c)	USEPA 1986b	
TAL	Metals	CLP SOW ILM02.0 (USEPA 1991)	CLP SOW ILM02.0 (USEPA 1991)	USEPA 1980	
	Cyanide	CLP SOW ILM02.0 (USEPA 1991)	CLP SOW ILM02.0 (USEPA 1991)	USEPA 1980 Hausknecht et al.	
	DIMP/DMMP	USAEC TT9	USAEC T8	USAEC T8 (mod)	
CSM degradation	IMPA/MPA	USAEC AAA9	USAEC UT02	USAEC UT02 (mod)	
products	Thiodiglycol	USAEC LW18	USAEC UW22	USAEC UW22 (mod)	
	1,4-dithiane	USAEC UL04	USAEC LL03	USAEC LL03 (mod)	
	Acetophenone	SW-846 8270A (USEPA 1986a)	SW-846 8270A (USEPA 1986a)	SW-846 8270A (USEPA 1986a)	
	Malononitrile	SW-846 8240A (USEPA 1986a)	SW-846 8240A (USEPA 1986a)	SW-846 8240A (USEPA 1986a)	
TCPU			Dennis (1983)		
	Chloride, sulfate, nitrate/nitrite	SM 4110 (APHA et al. 1989)	NA	NA	
	Total phosphorus	SM 2540 (APHA et al. 1989)	NA	NA	
	Alkalinity	SM 2320 (APHA et al. 1989)	NA	NA	
Water quality	Hardness	SM 2340 (APHA et al. 1989)	NA	NA	
	Total suspended solids	SM 2540 (APHA et al. 1989)	NA	NA	
	Biological oxygen demand	SM 5210 (APHA et al. 1989)	NA	NA	
	Chemical oxygen demand	SM 5220 (APHA et al. 1989)	NA	NA	

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TABLE 6-1 (continued) SUMMARY OF METHODS FOR CHEMICAL ANALYSIS

	Analyte	Aqueous Matrix	Solid Matrix	Biological Matrix
Water quality (cont.)	Ammonia	SM 4500 (APHA et al. 1989)	NA	NA
Organomer	curic compounds		ASTM E885-88 (1991e)	ASTM E885-88 (1991e)
Explos	sive analytes	USAEC LW12	USAEC UW14	USAEC UW14 (mod)
Dioxin/furans		Dioxin/furans CLP SOW DFLM01.0 (USEPA 1991a)		

No samples will be collected for this analysis.
 NA This parameter does not apply to the matrix.

CSM Chemical surety materials DIMP Diisopropylmethylphosphonate **DMMP** Dimethylmethylphosphonate **IMPA** isopropylmethylphosphonic acid MPA Methylphosphonic acid **PCBs** Polychlorinated biphenyls TAL Target analyte list TCL Target compound list TCPU bis(2,4,6-trichlorophenyl)urea **VOCs** Volatile organic compounds

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TABLE 6-2 SUMMARY OF METHODS FOR PHYSICAL TESTING OF SOIL/SEDIMENT SAMPLES

Analyte	Method	
USCS	ASTM D-2487	
Grain Size	ASTM D-422	
Total Organic Carbon	ASTM D-2974	
Percent Moisture	ASTM D-2216	

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TABLE 6-3 **SUMMARY OF METHODS FOR BIOASSAYS**

Species for Chronic Study	Method
Mysidopsis bahia	USEPA 1988
Hyalella azatec	Nebeker et al. 1984
Rana pipiens	Birge et al. 1985
Cyprinodon variegatus	USEPA 1988
Chironomus tentans	Nebeker et al. 1984
Daphnia magna	APHA 1985
Pimephales promelas	USEPA 1989b
Selenastrum capricornutum	USEPA 1989a

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TABLE 6-4 LIST OF TAL INORGANICS

Aluminum

Antimony

Arsenic

Barium

Beryllium

Cadmium

Calcium

Chromium

Cobalt

Copper

Cyanide

Iron

Lead

Magnesium

Manganese

Mercury

Nickel

Potassium

Selenium

Silver

Sodium

Thallium

Vanadium

Zinc

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using nitric acid and hydrogen peroxide. The digestate is subsequently analyzed by GFAA using the optimum instrumental conditions for the analytes of interest.

In order to obtain reporting limits lower than those provided by the ICAP method, mercury will be analyzed by CVAA. CVAA analysis is based on the absorption of radiation at 253.7 nm. A sample aliquot is initially digested with nitric acid to free any combined mercury. The mercury is then reduced to its elemental state and aerated from the solution into a closed system. The mercury vapor is passed through a cell positioned in the path of a mercury light source and the measured absorbance is proportional to the concentration of mercury in the sample.

b. Cyanide

Cyanide will be analyzed using a USEPA CLP method comparable to USEPA methods 335.2 and 335.3 (USEPA 1991b). The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a reflux-distillation operation and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined by volumetric titration or colorimetry. In the colorimetric measurement, the cyanide is converted to cyanogen chloride (CNCI) by reaction with chloramine-T at a pH of less than eight without hydrolyzing to the cyanate. After the reaction is complete, color is formed on the addition of pyridine-pyrazolone at 578 nm for pyridine-barbituric acid. To obtain colors of comparable intensity, the sample and the standards will contain the same salt content. The titrimetric measurement uses a standard solution of silver nitrate to titrate cyanide in the presence of a silver sensitive indicator.

6.6.1.2 Organic Analysis .

a. TCL Volatile Organic Compounds.

TCL volatiles listed in Table 6-5 will be analyzed using USEPA CLP methods (USEPA 1991c). The method involves purging of environmental sample and volatile organic-free water containing surrogates and internal standards with helium gas (following extraction). The purging chamber is heated to a predefined temperature and the vapor transferred to a sorbent tube which effectively traps the volatile organic compounds. The constituents are then backflushed onto a packed gas chromatographic column that is temperature programmed to separate the organic constituents. The volatile compounds are then detected using a mass spectrometer operating in the electron impact and full scan mode.

b. Semivolatile Organic Compounds

TCL Semivolatile Organic Compounds (SVOCs) listed in Table 6-6 will be analyzed using the USEPA CLP method. In accordance with this method, a one liter aliquot of sample will be acidified to pH 2 and extracted with methylene chloride using a continuous liquid-liquid extractor. The methylene chloride extract is dried and concentrated to 1.0 mL. The extract will be analyzed using a Gas Chromatograph and Mass Spectrometer. The compounds present will be identified by comparison of the sample mass spectrum to the mass spectrum of a standard of the suspected compound. A library search shall be executed for non-target sample components for tentative identification. Target components identified will be quantified based on the recovery of the internal standard with the nearest retention time.

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TABLE 6-5 LIST OF TCL VOLATILE ORGANIC COMPOUNDS

Acetone

Benzene

Bromodichloromethane

Bromoform

Bromomethane

2-Butanone

Carbon Disulfide

Carbon Tetrachloride

Chlorobenzene

Chloroethane

Chloroform

Chloromethane

Dibromochloromethane

1,1-Dichloroethane

1,1-Dichloroethene

1,2-Dichloroethane

1,2-Dichloroethene (total)

1,2-Dichloropropane

cis-1,3-Dichloropropene

trans-1,3-Dichloropropene

Ethylbenzene

2-Hexanone

Methylene Chloride

4-Methyl-2-pentanone

Styrene

1,1,2,2-Tetrachloroethane

Tetrachloroethene

Toluene

1,1,1-Trichloroethane

1,1,2-Trichloroethane

Trichloroethene

Vinyl Chloride

Xylene (Total)

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TABLE 6-6 LIST OF TCL SEMIVOLATILE ORGANIC COMPOUNDS

Acenaphthene

Acenaphthylene Anthracene

Benzo(a)anthracene Benzo(a)pyrene

Benzo(b)fluoranthene Benzo(g,h,i)perylene Benzo(k)fluoranthene

4-Bromophenyl-phenyl ether

Butylbenzylphthalate

bis(2-Chloroethoxyl)methane bis(2-Chloroethyl)ether bis(2-Chloroisopropyl)ether

bis(2-ethylhexyl)phthalate

Carbazole 4-Chloroaniline

4-Chloro-3-methylphenol 2-Chloronaphthalene

2-Chlorophenol

4-Chlorophenyl-phenyl ether

Chrysene

Dibenzo(a,h)anthracene

Dibenzofuran

1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
3,3'-Dichlorobenzidine
2,4-Dichlorophenol
Diethylphthalate
2,4-Dimethylphenol

Dimethylphthalate

Di-N-butylphthalate

Di-n-octylphthalate

4,6-Dinitro-2-methylphenol

2,4-Dinitrophenol 2,4-Dinitrotoluene 2,6-Dinitrotoluene Fluoranthene

Fluorene

Hexachlorobenzene Hexachlorobutadiene Hexachlorocyclopentadiene

Hexachloroethane

Indeno(1,2,3-cd)pyrene

Isophorone

2-Methylnaphthalene 2-Methylphenol

4-Methylphenol
Naphthalene
2-Nitroaniline
3-Nitroaniline

4-Nitroaniline Nitrobenzene 2-Nitrophenol 4-Nitrophenol

N-Nitroso-di-n-propylamine N-nitrosodiphenylamine Pentachlorophenol Phenanthrene

Phenol Pyrene

1,2,4-Trichlorobenzene 2,4,5-Trichlorophenol 2,4,6-Trichlorophenol

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b. TCL Pesticides/PCBs

Pesticides and PCBs listed in Table 6-7, will be analyzed by USEPA CLP method which employs use of gas chromatography with electron capture detector (GC/ECD) (USEPA 1991a). Identification of analytes is based on retention times and retention time data. Sample is extracted with hexane or methylene chloride. The methylene chloride extract is isolated, dried and concentrated after solvent substitution with methyl tert-butyl ether prior to analysis with GC. The solvent extract is then injected into a GC equipped with a linearized ECD for separation and analysis.

c. Explosives and Related Compounds

The following section describes the methodologies that will be used to analyzed samples for the explosives and related compounds listed in Table 6-8.

Nitroglycerin (NG) and pentaerythritol tetranitrate (PETN) will be analyzed in water using a USAEC method which employs RP-HPLC. The method for soil involves an initial extraction of the soil with acetonitrile in a sonic bath for two hours. Soil extracts are diluted 1/1 with aqueous CaCl₂ and filtered. The water samples are diluted with acetonitrile and filtered. Determination is by RP-HPLC on an LC-18 column (Supelco), using an eluent of 3/2 methanol-water at 1.5 mL/min, and UV detection at 220 nm.

All remaining explosives will be analyzed using HPLC (USAEC). The method employs solid phase extraction of 500 milliliters of an environmental aqueous sample or one gram of environmental solid sample using acetonitrile. The target analytes are separated by HPLC column using isocratic elution and detected using ultraviolet absorbance (UV) at 230 nanometers. USAEC certified methods are provided in Appendix D.

d. Chemical Surety Material (CSM) Degradation Products

The following section describes the methodologies that will be used to analyze samples for the chemical surety material degradation products and associated compounds as listed in Table 6-9. All USAEC certified methods are provided in Appendix D.

Diisopropylmethylphosphonate (DIMP) and dimethylmethylphosphonate (DMMP), breakdown products of the nerve agent GB, will be analyzed in aqueous and solid samples using GC-FPD via USAEC methods. A measured volume of sample or extract is directly injected onto the gas chromatographic column. Chromatographic conditions are described which permit the separation and measurement of DIMP and DMMP in environmental aqueous or solid samples. Qualitative identification is performed using retention times, and quantitative analysis is performed using standard curves. Isopropylmethylphosphonic acid (IMPA) and methylphosphonic acid (MPA) will be analyzed using a USAEC ion chromatography method.

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TABLE 6-7 LIST OF TCL PESTICIDES/PCBs

Aldrin

alpha-BHC

beta-BHC

delta-BHC

gamma-BHC (Lindane)

alpha-Chlordane

gamma-Chlordane

4,4'-DDD

4,4'-DDE

4,4'-DDT

Dieldrin

Endosulfan I

Endosulfan II

Liluosullari li

Endosulfan Sulfate

Endrin

Endrin aldehyde

Endrin ketone

Heptachlor

Heptachlor Epoxide

Methoxychior

Toxaphene

AROCLOR-1016

AROCLOR-1221

AROCLOR-1232

AROCLOR-1242

AROCLOR-1248

AROCLOR-1254

AROCLOR-1260

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TABLE 6-8 LIST OF EXPLOSIVES ANALYTES

Cyclotrimethylenetrinitramine (RDX)
Cyclotetramethylene tetranitramine (HMX)
1,3-Dinitrobenzene
2,4-Dinitrotoluene
2,6-Dinitrotoluene
N-Methyl-N,2,4,6-Tetranitrobenzoaniline (TETRYL)
Nitrobenzene
Nitroglycerin
Pentaerythritol tetranitrate (PETN)
1,3,5,-Trinitrobenzene
2,4,6-Trinitrotoluene

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TABLE 6-9 LIST OF CHEMICAL SURETY MATERIAL DEGRADATION PRODUCTS AND ASSOCIATED COMPOUNDS

CSM Degradation Products

Diisopropylmethylphosphonate (DIMP)

1,4-Dithiane

1,4-Oxythiane

Thiodiglycol

p-Chlorophenylmethylsulfoxide

p-Chlorophenylmethylsulfone

p-Chlorophenylmethylsulfide

Isopropylmethylphosphonic acid (IMPA)

Methylphosphonic acid (MPA)
Associated Compounds

bis(2,4,6-trichlorophenyl)urea (TCPU)

Dimethylmethylphosphonate (DMMP)

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TABLE 6-10 LIST OF DIOXINS AND FURANS

1,2,3,4,6,7,8-Heptachlorodibenzofuran 1,2,3,4,7,8,9-Heptachlorodibenzofuran 1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin 1,2,3,4,7,8-Hexachlorodibenzofuran 1,2,3,6,7,8-Hexachlorodibenzofuran 2,3,4,6,7,8-Hexachlorodibenzofuran 1,2,3,7,8,9-Hexachlorodibenzofuran 1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin 1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin 1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin Octachlorodibenzofuran Octochlorodibenzo-p-dioxin 1,2,3,7,8-Pentachlorodibenzofuran 2,3,4,7,8-Pentachlorodibenzofuran 1,2,3,7,8-Pentachlorodibenzo-p-dioxin 2,3,7,8-Tetrachlorodibenzofuran 2,3,7,8-Tetrachlorodibenzo-p-dioxin

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TABLE 6-11 LIST OF WATER QUALITY PARAMETERS

Alkalinity
Ammonia
Biochemical Oxygen Demand (BOD)
Chemical Oxygen Demand (COD)
Chloride
Hardness
Nitrate/Nitrite
Sulfate
Total Organic Carbon (TOC)
Total Phosphorus
Total Suspended Solids

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Total phosphorus will be analyzed by autoanalyzer in aqueous and solid samples by ascorbic acid method-SM Method 4500 (APHA et al. 1989). The persulfate digestion method will be used prior to analysis, which includes the heating of the sample in the presence of sulfuric acid and ammonium persulfate for 30 to 40 minutes. All forms of phosphate are converted to orthophosphate and the concentration is determined colorimetrically. Ammonium molybdate and potassium antimonyl tartrate react in an acid medium with dilute solutions of ortho-phosphate to form a heteropoly acid phosphomolybdic acid. This complex is reduced to an intensely blue-colored complex by ascorbic acid. The color is proportional to the orthophosphate concentration.

Alkalinity will be analyzed by the titration method-SM Method 2320 (APHA et al. 1989). This method uses the volume of standard acid required to titrate a portion of the sample to a designated pH to extrapolate an alkalinity value.

Hardness will be analyzed by the EDTA (ethylenediaminetetraacetic acid and its sodium salts) titrimetric method-SM Method 2340 (APHA et al. 1989). This method measures the calcium and magnesium ions by the addition of EDTA to the sample which creates a color change when all calcium and magnesium have been complexed. A calculation provides the hardness value.

Total suspended solids (TSS) will be analyzed by SM Method 2540 (APHA et al. 1989). The method involves the drying of samples at 103-105°C on a filter. The increase in weight of the filter represents the value of TSS present in the sample.

Biochemical oxygen demand (BOD) will be determined using SM Method 5210 (APHA et al. 1989). The method involves measuring the oxygen utilized during an incubation period of 5 days, following dilution. Dissolved oxygen (DO) is measured initially and after incubation, and the BOD is computed from the difference between initial and final DO.

Chemical oxygen demand (COD) will be determined using SM Method 5220 (APHA et al. 1989). The COD is used as a measure of the oxygen equivalent of the organic matter content of a sample that is susceptible to oxidation by a strong chemical oxidant. A sample is refluxed in a strongly acidic solution with a known excess of potassium dichromate. After digestion, the remaining unreduced potassium dichromate is titrated with ferrous ammonium sulfate to determine the amount of potassium dichromate consumed and the oxidizable organic matter is calculated in terms of oxygen equivalent.

In the determination of total organic carbon (TOC) by SM Method 5310 (APHA et al. 1989), organic molecules are broken down to single carbon units, converted to a single molecular form and then converted to carbon dioxide. The carbon dioxide may be measured directly by a nondispersive infrared analyzer, it may be reduced to methane and measured with a flame ionization detector, or the carbon dioxide may be titrated chemically. The exact method will be determined by the concentration of TOC expected in the sample. The persulfate-ultraviolet method is useful for the determination of trace levels of TOC and the wet-oxidation method is suitable only for water containing at least 0.1 mg nonpurgeable organic carbon per liter. If concentrations of TOC exceed 1 mg/L, a combustion infrared method will be used.

Ammonia will be determined using the ammonia-selective electrode method-SM Method 4500 (APHA et al. 1989), which uses a hydrophobic gas-permeable membrane to separate the sample solution from an electrode internal solution of ammonium chloride. Dissolved ammonia ($NH_{3(aq)}$) and

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NH₄⁺) is converted to NH₃ by raising the pH to above 11 with a strong base. NH₃ diffuses through the membrane and the internal solution pH is sensed by a pH electrode. The fixed level of chloride in the internal solution is sensed by a chloride ion-selective electrode that serves as the reference electrode. Potentiometric measurements are made with a pH meter having an expanded millivolt scale or with a specific ion meter.

6.6.2 Physical Analyses for Solid Samples

Soil and sediment samples will be collected and analyzed for selected physical analysis by ASTM Methods. Analyses include percent moisture/percent solids, grain size distribution, total organic carbon, and United Soil Classification System (USCS) designation, also listed in Table 6-12.

Percent moisture in soil will be determined using ASTM Method D-2216 (1991b). This method involves the determination of the percent water mass in a known mass of undried soil by weighing the soil before and after drying in an oven controlled at 110°C. The water content of a material is defined as the ratio, expressed as a percentage, of the mass of "pore" or "free" water in a given mass of material to the mass of the solid material particles. Percent solids will be determined in sediment as opposed to percent moisture. A procedure in the CLP Method for organics analysis will be employed.

Grain size distribution will be determined using ASTM Method D-442 (1991a). This method covers the quantitative determination of the distribution of particle sizes in soil. A No. 200 sieve is used to separate particles larger than 75µm from the soil, while the distribution of particles smaller than 75µm is determined by a sedimentation process, using a hydrometer to secure the necessary data.

Organic content of soils will be determined using ASTM Method D-2974 (1991d). This method involves the ignition of an oven dried soil sample in a muffle furnace. The weight of the sample is taken before and after ignition, and the organic mass is the difference of the two masses. The organic content (a percentage) is expressed as this difference divided by the weight of the sample before ignition.

Classification of soils will be based on laboratory determination of particle-size characteristics, liquid limit, and plasticity index using ASTM Method D-2487 (1991c). The system is based on the Unified Soil Classification System (USCS).

6.6.3 Methods for the Chemical Analysis of Tissue Samples

Methods used for the chemical analysis of tissue samples have been modified from standardized methods used to analyze solid matrices. Modifications include initial preparation of samples and additional cleanup steps to eliminate matrix interferences.

6.6.3.1 Initial Laboratory Preparation of Samples. The samples will be received by the laboratory and logged in to the sample tracking system. Biological tissue samples will be securely stored in a freezer at -20°F from the time of receipt through final analysis.

To prepare the biological organism, tissue, or organ sample for analysis of organics compounds, the sample will be unwrapped and weighed. The samples will be chopped into 3 cm chunks using a scalpel or sharp knife and mallet. Crushed or pelleted dry ice will be ground in a

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TABLE 6-12 LIST OF PHYSICAL TESTING PARAMETERS

USCS Classification Percent Moisture Total Organic Carbon Grain Size Distribution

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micro-grinder to precool the grinder. Tissue samples will then be homogenized to a uniform consistency. Samples for non-volatile analyses will be ground and thoroughly mixed two more times. The grinder will be cleaned of any remaining material, which will be added to the ground sample. The sample will be transferred to an appropriately solvent-cleaned glass jar with aluminum or teflon lined plastic lid, and stored in a freezer at -20°F for later subsampling and analysis.

An aliquot of the ground biological sample, as prepared above in a micro-grinder, may be used for the analysis of inorganics. However, the stainless steel grinder may be a possible source of nickel and chromium contamination. Therefore, samples will be prepared for analysis of inorganics using a blender equipped with a tantalum blade or other equipment that has been proven contaminant-free. A fish that weighs between 50 and 300 grams will be chopped with a meat cleaver or knife and mallet and placed into a blender that has been pre-cooled with dry ice. The sample will be blended with dry ice until homogenous, and will be stored in a loosely sealed plastic bag (to allow carbon dioxide to escape) and frozen for at least 16 hours before digestion.

Preparation of plant material samples generally requires grinding into a paste or drying and grinding prior to extraction and analysis.

6.6.3.2 Inorganic Chemical Analyses for Biotic Samples. USEPA TAL inorganics in biotic tissues and in plant material will be analyzed using one of the following methodologies: conventional direct aspiration flame atomic absorption spectroscopy (FAA), graphite furnace atomic absorption spectroscopy (GFAA), cold vapor atomic absorption (CVAA), hydride generation atomic absorption (HYDAA), inductively coupled argon plasma emission spectroscopy (ICAP), or automated spectrophotometry. TAL metals will be analyzed in tissue using USEPA (1980) and cyanide using Hausknecht et al. (no date).

a. Antimony, Beryllium, Cadmium, Chromium, Copper, Lead, Mercury, Nickel, Silver, Thallium, and Zinc

Samples prepared for the analysis of antimony, beryllium, cadmium, chromium, copper, lead, mercury, nickel, silver, thallium, and zinc will undergo wet digestion procedures. A 5 gram aliquot of the homogenized tissue is mixed with concentrated nitric acid for 15 hours and then gently heated in increasing increments to a maximum of 250°C. The sample is digested until all tissue has been solubilized. The sample is then oxidized with perchloric acid over heat until the solution is clear. Analysis for arsenic, lead, and selenium require the addition of a matrix modifier during the digestion procedure.

Samples prepared for the analysis of cadmium, copper, and zinc are analyzed by direct aspiration FAA. The sensitivity of FAA is adequate for these analytes due to the relatively high expected concentrations in biological tissues.

In order to obtain quantitation limits lower than those provided by conventional FAA methods, and because concentrations are expected to be low in biological tissues, beryllium, chromium, lead, nickel, silver, and thallium will be analyzed using GFAA, and antimony, arsenic, and selenium will be analyzed using HYDAA.

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Total and inorganic mercury will be analyzed using CVAA. Organic mercury concentrations will be estimated from the total and inorganic concentrations. The CVAA analysis is based on the absorption of radiation at 253.7 nm by mercury vapor. Combined mercury is freed during the digestion. The mercury is then reduced to its elemental state and aerated from the solution into a closed system. The mercury vapor is passed through a cell positioned in the path of a mercury light source and the measured absorbance is proportional to the concentration of mercury in the sample.

b. Aluminum, Barium, Cobalt, Manganese, and Vanadium

The ICAP method will be used for inorganic analytes not amenable to analysis by atomic absorption (e.g. aluminum, barium, cobalt, manganese, and vanadium). The method is similar to USEPA CLP-M method 200.7. An aliquot of homogenized sample is mixed with concentrated nitric acid and left to digest overnight. The sample is then refluxed without boiling until digestion is complete. Following digestion, the sample is evaporated, and heated with nitric acid and hydrogen peroxide until no further oxidation is evident. The samples are then diluted and analyzed. Analysis by ICAP involves the measurement of atomic emission by optical spectrometry. The ICAP source consists of a flowing stream of argon gas ionized by an applied radio frequency field which is coupled to the ionized gas by a water-cooled coil surrounding a quartz torch. Characteristic atomic line emission spectra are produced by the radio-frequency inductively coupled plasma.

c. Cyanide

Cyanide will be analyzed in tissue samples using a spectrophotometric method. The method is comparable to USEPA Method 335.2. A weighed aliquot of the homogenized sample is blended with deionized distilled water. The blended sample is transferred to a boiling flask and connected to a condenser, absorber, and trap in a distillation train. The sample is refluxed with concentrated sulfuric acid and magnesium chloride solution. Hydrogen cyanide is released from the solution and absorbed into sodium hydroxide. The cyanide ion is then determined colorimetrically at 570 nm with an automated spectrophotometer.

6.6.3.3 Organic Chemical Analyses for Biotic Samples. Clean-up of biological tissues for analysis of organic chemicals includes column chromatography methods. For organisms, tissue, and organ samples, the first step in the clean-up is gel-permeation chromatography (GPC), which is used for removal of lipids. Clean-up for analysis of other organic compounds may include additional methods such as florisil, silica gel, or alumina column chromatography for removal of polar biogenic compounds.

The organic constituents will be analyzed by gas chromatography/mass spectroscopy (GC/MS), gas chromatography-electron capture detector (GC-ECD), gas chromatography-flame photometric detection (GC-FPD), high pressure liquid chromatography (HPLC), ion chromatography (IC), and mobile phase ion chromatography (MPIC). The following identifies and describes the methods that will be used for the analysis of organic compounds:

a. TCL Semivolatile Organic Compounds

Analysis of TCL semivolatile organic compounds in biological samples will be analyzed by GC/MS. A homogenized tissue sample undergoes Soxhlet extraction with sodium sulfate, methanol and

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dichloromethane for 24 hours. After Soxhlet extraction, the sample undergoes liquid-liquid aqueous extractions in order to remove water and methanol and to enhance the partitioning of organic compounds. The extract is dried over sodium sulfate, concentrated and loaded onto a calibrated gel permeation chromatography (GPC) column for removal of biological macromolecules such as lipids. The collected extract is then analyzed with GC/MS operated in the electron impact mode.

b. TCL Pesticides/PCBs

Samples for organochlorine pesticides and polychlorinated biphenyls analyses will undergo the same extraction procedures used for analysis of semivolatile compounds. In addition, the sample will undergo clean-up using alumina column chromatography. The collected extract is then analyzed with GC/ECD.

c. Explosives and Related Compounds

Explosives in organisms, tissues, organs, and plant materials will be analyzed using HPLC. A 0.5 gram aliquot of dried, homogenized sample will be digested in hydrochloric acid, and then extracted with diethyl ether. This extract, dried and dissolved in methylene chloride, is loaded onto a florisil solid phase extraction cartridge and eluted with ethyl acetate. The sample is then filtered and the analytes are separated by a reverse-phase HPLC column using isocratic elution and detected using ultraviolet absorbance (UV). The method does not require the addition of GPC cleanup for the removal of lipids because lipids do not interfere in HPLC analyses.

d. Chemical Surety Material (CSM) Degradation Products

CSM degradation products will be analyzed using modified accepted methods for analysis of compounds in soil and sediment, or other published methods. The methods will be modified by the addition of clean-up techniques when necessary.

Thiodiglycol will be analyzed with HPLC-EC/UV. The homogenized tissue sample is extracted with alkaline methanol on a wrist-action shaker. A portion of the methanol is filtered, and removed by evaporation under a nitrogen stream. The extract is acidified and buffered, and brought to volume with water. Liquid chromatographic conditions described in the method permit the separation and measurement of the thiodiglycol in the extract. Analyte identification is performed using retention times, and quantitative analysis is performed using a standard curve of area counts.

Organosulfur compounds will be analyzed using modified USAEC methodologies. The homogenized tissue sample is extracted with chloroform by ultrasonic vibration. The extract is cleaned-up on a GPC column and then separated by gas chromatography. Detection is by flame-photometry.

DIMP and DMMP will be analyzed with GC-FPD and HPLC-UV, respectively. IMPA and MPA will be analyzed using a USAEC method utilizing ion chromatography (IC). Because the extraction will be aqueous, the addition of a GPC clean-up step is unnecessary.

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e. Organomercuric Compounds

Organomercuric compounds will be analyzed using ASTM Method E 885-88 which employs CVAA (ASTM 1991e). Addition of potassium permanganate is followed by a persulfate oxidation step to ensure that organo-mercury compounds, if present, will be oxidized to the mercuric ion prior to measurement. A heat step is required for methyl mercuric chloride when present in or spiked to a natural system. Determination is based on the absorption of radiation at 253.7 nm by mercury vapor. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

6.6.3.4 Other Analyses.

a. Lipid Content

Percent lipids in biological tissues will be analyzed by first extracting a homogenized tissue sample in a Soxhlet extractor with methanol and dichloromethane. The extract is then dried over sodium sulfate, concentrated and loaded onto a calibrated gel permeation chromatography (GPC) column. Biological macromolecules elute in the dump phase, or first 100 mL of eluate. This fraction is dried under nitrogen and air dried on an aluminum planchet. Lipid content is determined gravimetrically.

b. Dry Weight Determination

A homogenized tissue sample is dried in a preweighed drying dish overnight. The solid residue is weighed to determine the dry weight. Dry weight is determined whenever results will be reported as dry weight concentrations.

6.6.4 Bioassay Methods

6.6.4.1 Surface Water Chronic Bloassays. Surface water chronic bioassays to be performed as part of the TERA include *Cyprinodon variegatus* (USEPA 1988), *Daphnia magna* (APHA et al. 1989), *Mysidopsis bahia* (USEPA 1988), *Pimephales promelas* (USEPA 1989b), *Rana pipiens* (Birge et al. 1985), and *Selanastrum capricornutum* (USEPA 1989a).

In the estuarine fish bioassay, larvae of the sheepshead minnow *C. variegatus* (less than 24 hours old) are placed into test cups containing surface water samples, reference site water, and laboratory control waters. *C. variegatus* are checked daily, at which time the number of dead organisms are counted and removed and the remaining animals fed. The test is terminated after 7 days. *C. variegatus* survival and weight in the surface water, the reference site water, and the laboratory control waters will be compared to determine if there is a statistically significant difference.

In the freshwater zooplankton bioassay, neonate *D. magna* are placed into beakers containing surface water samples, reference site water, and laboratory control waters. *D. magna* are observed daily, at which time the number of dead organisms and any neonates are counted and removed. Feeding occurs on alternate days. The test is terminated after six broods are produced by the control animals (approximately 21 days). *D. magna* survival and reproduction in the surface water, reference

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site water, and laboratory control waters will be compared to determine if there is a statistically significant difference.

In the estuarine invertebrate bioassay, *M. bahia* (7 days old) are placed into test cups containing surface water samples, reference site water, and laboratory control waters. *M. bahia* are checked daily, at which time the number of dead organisms are counted and removed and the remaining animals are fed. The test is terminated after 7 days. *M. bahia* survival, reproduction, and fecundity (measured as the percentage of females with eggs in the oviduct and/or brood pouch) in the surface water, the reference site water, and laboratory control waters will be compared to determine if there is a statistically significant difference.

In the freshwater fish bioassay, embryos of the fathead minnow *P. promelas* are placed into test cups containing surface water samples, reference site water, and laboratory control waters. After hatching, the larvae are fed. The number of live and dead organisms in each test chamber are noted regularly. After 7 days, the test is terminated and the number of surviving larvae are counted. Survival and teratogenic effects in test water, the reference site water, and laboratory control groups will be compared to determine if there is a statistically significant difference.

In the amphibian bioassay, fertilized *R. pipiens* eggs are placed into cups containing surface water samples, reference site water, and laboratory control waters. Teratogenic effects are determined when the larvae hatch. The number of surviving larvae is determined at the termination of the assay, generally 4-8 days after the larvae hatch. Amphibian survival in the surface water and the reference site and laboratory control waters will be compared to determine if there is a statistically significant difference.

In the phytoplankton bioassay, a known number of *S. capricornutum* cells are added to beakers containing surface water samples, reference site water, and laboratory control waters. After incubating the algal-test solution combination for 96 hours, algal growth is determined by measuring changes in cell density (cells/ml), biomass, or chlorophyll *a* content. At a minimum, both biomass and chlorophyll *a* content will be used as measures of algal growth. Employing several measures of algal growth is recommended because algal response varies with the type of toxicant (Sirois 1990). Growth of algae in the surface water, the reference site water, and laboratory control waters will be compared to determine if there is a statistically significant difference.

6.6.4.2 Sediment Chronic Bioassays. Sediment chronic bioassays to be performed as part of the TERA include *Chironomus tentans* (Nebeker et al. 1988) and *Hyalella azteca* (Nebeker et al. 1988).

In the freshwater benthic invertebrate bioassay, test sediment, reference site sediment, and laboratory control sediment are placed into beakers and covered with water. After the sediments have settled completely, 10 day old instar *C. tentans* larvae are introduced into the water. *C. tentans* are fed during this test. The test is terminated after 15 days at which time the survivors are counted and weights are measured. *C. tentans* survival and weight in the test sediment, the reference site sediment, and the laboratory control sediments will be compared to determine if there is a statistically significant difference.

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In the estuarine benthic invertebrate bioassay, test sediment, reference site sediment, and laboratory control sediment are placed into beakers and covered with water. After the sediments have settled completely, *H. azteca* are introduced into the water. *H. azteca* are fed during the partial life cycle test, which is terminated after 28 days. Survivors are counted at the time the test is terminated. Adult and neonate survival in the test sediment and the reference site and laboratory control sediments will be compared to determine if there is a statistically significant difference.

6.7 USEPA CLP CONTRACT REQUIRED QUANTITATION AND DETECTION LIMITS

The USEPA CLP has established certified quantitation limits (CRQL/CRDL), set forth in the SOW. The CRDL is the minimum sample quantitation limit (SQL) acceptable under the contract SOW. The sample quantitation limit is the method detection limit adjusted for sample size and matrix effects (e.g. percent moisture). The Contract Required Quantitation Limits, are provided in Tables 6-16 through 6-20 for volatile, semivolatile, and pesticide/PCB organic compounds, inorganics, and dioxins/furans. Tissue detection limits will be based on detection limits for soil/sediment methodologies while any dilution factors will be accounted for in the sample quantitation. ESE method specific reporting limits are provided in the ESE Quality Assurance Manual provided in Appendix E.

6.8 ASTM METHOD DETECTION LIMITS

ESE's method detection limits for all analyses using ASTM methodologies are presented in Table 6-18.

6.9 USAEC CERTIFIED AND UPPER REPORTING LIMITS

The lowest concentration that is reported for any analyte has been established in the USAEC program from a statistical analysis of spikes and blanks. The concentration, termed the certified reporting limit (CRL), is the lowest value that can be reported with a 90% confidence level. The upper reporting limit (URL) for the certified range is the highest standard analyzed during the method certification. ESE's CRLs and URLs are presented in Table 6-19 (explosives) and Table 6-20 (CSM degradation products.

6.10 REFERENCE MATERIALS

Reference standards are required to generate certification data, calibrate instruments, spike analytical surrogates or standards, and prepare QC samples. These solutions must be of known concentration and purity to achieve the criteria necessary for validation of analytical analyses.

Standards used to conduct analytical analyses will be either Standard Analytical Reference Materials (SARMs) or Interim Reference Materials (IRMs). SARMs are developed and distributed by the National Institute of Standards and Technology (NIST) or by National Bureau of Standards (NBS). SARMs are the preferred standard because IRMs are not as rigorously characterized.

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TABLE 6-13 CLP CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs) FOR TCL VOLATILE ORGANIC COMPOUNDS

Anabaa	USAEC	CRQLs			
Analyte	Acronym	Solid-Low conc. (mg/kg) ^a	Aqueous (μg/L)		
Methylene chloride	CH2CL2	0.01	10.0		
1,1-Dichlororethane	11DCLE	0.01	10.0		
trans-1,2-Dichloroethene	12DCE	0.01	10.0		
1,1-Dichloroethylene	11DCE	0.01	10.0		
Chloroform	CHCL3	0.01	10.0		
1,2-Dichloroethane	12DCLE	0.01	10.0		
1,1,1-Trichloroethane	111TCE	0.01	10.0		
Carbon tetrachloride	CCL4	0.01	10.0		
Trichloroethylene	TRCLE	0.01	10.0		
Benzene	C6H6	0.01	10.0		
1,1,2-Trichloroethane	112TCE	0.01	10.0		
Tetrachloroethylene	TCLEE	0.01	10.0		
Toluene	MEC6H5	0.01	10.0		
Chlorobenzene	CLC6H5	0.01	10.0		
Ethylbenzene	ETC6H5	0.01	10.0		
1,2-Dichloropropane	12DCLP	0.01	10.0		
cis-1,3-Dichloropropylene	C13DCP	0.01	10.0		
Vinyl chloride	C2H3CL	0.01	10.0		
Chloroethane	C2H5CL	0.01	10.0		
Chloromethane	CH3CL	0.01	10.0		
Bromoform	CHBR3	0.01	10.0		
Dibromochloromethane	DBRCLM	0.01	10.0		
trans-1,3-Dichloropropene	T13DCP	0.01	10.0		
1,1,2,2-Tetrachloroethane	TCLEA	0.01	10.0		
Bromodichloromethane	BRDCLM	0.01	10.0		
Bromomethane	CH3BR	0.01	10.0		
Acetone	ACET	0.01	10.0		
Carbon disulfide	CS2	0.01	10.0		
2-Butanone	MEK	0.01	10.0		
4-Methyl-2-pentanone	MIBK	0.01	10.0		
Styrene	STYR	0.01	10.0		
Xylene	XYLEN	0.01	10.0		

Quantitation limits listed for solids are based on wet weight. The quantitation limits calculated by the laboratory, calculated on dry weight basis as required by contract, will be higher.

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TABLE 6-14 CLP CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs) FOR TCL SEMI-VOLATILE ORGANIC COMPOUNDS

A	USAEC	CRQLs			
Analyte	Acronym	Solid (mg/kg) ^a	Aqueous (μg/L)		
Phenol	PHENOL	0.33	10		
Bis(2-chloroethyl)ether	B2CLEE	0.33	10		
2-Chlorophenol	2CLP	0.33	10		
1,3-Dichlorobenzene	13DCLB	0.33	10		
1,4-Dichlorobenzene	14DCLB	0.33	10		
1,2-Dichlorobenzene	12DCLB	0.33	10		
2-Methylphenol	2MP	0.33	10		
Bis(2-chloroisopropyl)ether	B2CIPE	0.33	10		
4-Methylphenol	4MP	0.33	10		
N-Nitroso-di-n-propylamine	NNDNPA	0.33	10		
Hexachloroethane	CL6ET	0.33	10		
Nitrobenzene	NB	0.33	10		
Isophorone	ISOPHR	0.33	10		
2-Nitrophenol	2NP	0.33	10		
2,4-Dimethylphenol	24DMPN	0.33	10		
Bis(2-chloroethoxy)methane	B2CEXM	0.33	10		
2,4-Dichlorophenol	24DCLP	0.33	10		
1,2,4-Trichlorobenzene	124TCB	0.33	10		
Naphthalene	NAP	0.33	10		
4-Chloroaniline	4CANIL	0.33	10		
Hexachlorobutadiene	HCBD	0.33	10		
4-Chloro-3-methylphenol	4CL3C	0.33	10		
2-Methylnaphthalene	2MNAP	0.33	10		
Hexachlorocyclopentadiene	CL6CP	0.33	10		
2,4,6-Trichlorophenol	246TCP	0.33	10		
2,4,5-Trichlorophenol	245TCP	0.80	25		
2-Chloronaphthalene	2CNAP	0.33	10		
2-Nitroaniline	2ANIL	0.80	25		
Dimethylphthalate	DMP	0.33	10		
Acenaphthylene	ANAPYL	0.33	10		
2,6-Dinitrotoluene	26DNT	0.33	10		
3-Nitroaniline	3NANIL	0.80	25		
Acenaphthene	ANAPNE	0.33	10		
2,4-Dinitrophenol	24DNP	0.80	25		

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TABLE 6-14 (continued) CLP CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs) FOR TCL SEMI-VOLATILE ORGANIC COMPOUNDS

Harris and American Company	USAEC	CRQLs		
Analyte	Acronym	Solid (mg/kg) ^a	Aqueous (μg/L)	
4-Nitrophenol	4NP	0.80	25	
Dibenzofuran	FURANS	0.33	10	
2,4-Dinitrotoluene	24DNT	0.33	10	
Diethylphthalate	DEP	0.33	10	
4-Chlorophenyl-phenylether	4CLPPE	0.33	10	
Fluorene	FLRENE	0.33	10	
4-Nitroaniline	4NANIL	0.80	10	
4,6-Dinitro-2-methylphenol	46DN2C	0.80	10	
N-Nitrosodiphenylamine	NNDPA	0.33	10	
4-Bromophenyl-phenyl ether	4BRPPE	0.33	10	
Hexachlorobenzene	CL6BZ	0.33	10	
Pentachlorphenol	PCP	0.80	10	
Phenanthrene	PHANTR	0.33	10	
Anthracene	ANTRC	0.33	10	
Di-n-butylphthalate	DNBP	0.33	10	
Fluoranthene	FANT	0.33	10	
Pyrene	PYR	0.33	10	
Butylbenzylphthalate	BBZP	0.33	10	
3,3'-Dichlorobenzidine	33DCBD	0.33	10	
Benz[a]anthracene	BAANTR	0.33	10	
Chrysene	CHRY	0.33	10	
Bis(2-ethylhexyl)phthalate	B2EHP	0.33	10	
Di-n-octylphthalate	DNOP	0.33	10	
Benzo[b]fluoranthene	BBFAN	0.33	10	
Benzo[k]fluoranthene	BKFANT	0.33	10	
Benzo[a]pyrene	BAPYR	0.33	10	
Indeno(1,2,3-cd)pyrene	ICDPR	0.33	10	
Dibenz[a,h]anthracene	DBAHA	0.33	10	
Benzo[g,h,i]perylene	BGHIPY	0.33	10	

Quantitation limits for solids are based on wet weight. The quantitation limits calculated by the laboratory, calculated on dry weight basis as required by the contract, will be higher.

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TABLE 6-15
CLP CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs) FOR TAL INORGANICS*

Analyte	USAEC Acronym	CRDL		
Atlatyte	Solid (mg/kg)		Aqueous (μg/L)	
Aluminum	AL	40	200	
Antimony	SB	12	60	
Arsenic	AS	2	10	
Barium	ВА	40	200	
Beryllium	BE	1	5	
Cadmium	CD	1	5	
Calcium	CA	1,000	5,000	
Chromium	CR	2	10	
Cobalt	CO	10	50	
Copper	CU	5	25	
Cyanide	CN	2	10	
Iron	FE	20	100	
Lead	PB	1	3	
Magnesium	MG	1,000	5,000	
Manganese	MN	3	15	
Mercury	HG	0.1	0.2	
Nickel	NI	8	40	
Potassium	K	1,000	5,000	
Selenium	SE	1	5	
Silver	AG	2	10	
Sodium	NA	1,000	5,000	
Thallium	TL	2	10	
Vanadium	V	10	50	
Zinc	ZN	4	20	

The 1991 CLP SOW does not state CRQLs for inorganics. These CRDLs presented for aqueous samples are representative only of the ICAP analysis, because the CRQLs for others are not specified. The CRDLs presented for soil originated from the 1987 CLP SOW and represent ICAP, with the exception of: GFAA for As, Se, and Pb; CVAA for Hg; and autoanalyzer for cyanide.

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TABLE 6-16 CLP CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs) FOR TCL PESTICIDES/PCBs

		CRQLs			
Analyte	USAEC Acronym	Solid (mg/kg)*	Aqueous (μg/L)		
alpha-BHC	ABHC	0.0017	0.05		
beta-BHC	BBHC	0.0017	0.05		
delta-BHC	DBHC	0.0017	0.05		
gamma-BHC (Lindane)	LIN	0.0017	0.05		
Heptachlor	HPLC	0.0017	0.05		
Aldrin	ALDRN	0.0017	0.05		
Heptachlor epoxide	HPCLE	0.0017	0.05		
Endosulfan I	AENSLF	0.0017	0.05		
Dieldrin	DLDRN	0.0033	0.10		
4,4'-DDE	PPDDE	0.0033	0.10		
Endrin	ENDRN	0.0033	0.10		
Endosulfan II	BENSLF	0.0033	0.10		
4,4'-DDD	PPDDD	0.0033	0.10		
Endosulfan sulfate	ESFSO4	0.0033	0.10		
4,4'-DDT	PDDDT	0.0033	0.10		
Endrin ketone	ENDRNK	0.0033	0.10		
Methoxychlor	MEXCLR	0.017	0.50		
Endrin aldehyde	ENDRNA	0.033	0.10		
alpha-Chlordane	ACLDAN	0.033	0.05		
gamma-Chlordane	GCLDAN	0.067	0.05		
Toxaphene	TXPHEN	0.033	5.0		
AROCLOR-1016	PCB016	0.033	1.0		
AROCLOR-1221	PCB021	0.033	2.0		
AROCLOR-1232	PCB232	0.033	1.0		
AROCLOR-1242	PCB242	0.033	1.0		
AROCLOR-1248	PCB248	0.033	1.0		
AROCLOR-1254	PCB254	0.033	1.0		
AROCLOR-1260	PCB260	0.033	1.0		

Quantitation limits listed for solids are based on wet weight. The quantitation limits calculated by the laboratory, calculated on dry weight basis as required by the contract, will be higher.

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TABLE 6-17 CLP CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs) FOR DIOXINS AND FURANS

	CRQLs"		
Analyte	Solid (mg/kg)	Aqueous (ng/L)	
2,3,7,8-Tetrachlorinated dibenzo-p-dioxin	0.001	0.01	
2,3,7,8-Tetrachlorinated dibenzofuran	0.001	0.01	
1,2,3,7,8-Pentachlorinated dibenzofuran	0.0025	0.025	
1,2,3,7,8-Pentachlorinated dibenzo-p-dioxin	0.0025	0.025	
2,3,4,7,8-Pentachlorinated dibenzofuran	0.0025	0.025	
1,2,3,4,7,8-Hexachlorinated dibenzofuran	0.0025	0.025	
1,2,3,6,7,8-Hexachlorinated dibenzofuran	0.0025	0.025	
1,2,3,4,7,8-Hexachlorinated dibenzo-p-dioxin	0.0025	0.025	
1,2,3,6,7,8-Hexachlorinated dibenzo-p-dioxin	0.0025	0.025	
1,2,3,7,8,9-Hexachlorinated dibenzo-p-dioxin	0.0025	0.025	
2,3,4,6,7,8-Hexachlorinated dibenzofuran	0.0025	0.025	
1,2,3,7,8,9-Hexachlorinated dibenzofuran	0.0025	0.025	
1,2,3,4,6,7,8-Heptachlorinated dibenzofuran	0.0025	0.025	
1,2,3,4,6,7,8-Heptachlorinated dibenzo-p-dioxin	0.0025	0.025	
1,2,3,4,7,8,9-Heptachlorinated dibenzofuran	0.0025	0.025	
Octachlorinated dibenzo-p-dioxin	0.005	0.050	
Octachlorinated dibenzofuran	0.005	0.050	

In addition, data are reported for the total concentration of all detected polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans. However, because the number of non-2,3,7,8-substituted isomers that might be detected in a sample is unpredictable, it is not possible to assign CRQL values to the total homologue concentrations.

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TABLE 6-18 ESE METHOD DETECTION LIMITS (MDLs) FOR WATER QUALITY ANALYTES

Analysis (1997)	AICAEC Assesses	MDL	
Analyte	USAEC Acronym	Aqueous (μg/L)	
Ammonium	NH4	20	
Bicarbonate (Alkalinity)	НСОЗ	5,000	
Bromide	BR	100	
Carbonate (Alkalinity)	CO3	5,000	
Chemical Oxygen Demand	COD	5,000	
Chloride	CL	500	
Fluoride	F	100	
lodine	f	500	
Nitrate	NO3	10	
Nitrite	NO2	10	
Phosphate	PO4	10	
Sulfate	SO4	500	
Sulfide	SULFID	500	
Total Dissolved Solids	TDS	10,000	
Total Suspended Solids	TSS	4,000	

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TABLE 6-19 ESE CERTIFIED REPORTING LIMITS FOR EXPLOSIVES

Analyte	USAEC Acronym	Aqueous Reporting Limits (µg/L)		Solid Reporting Limits (µg/g)	
e di 1977 e di Tomano di 1995 de 1988. Per		Certified	Upper	Certified	Upper
Cyclotrimethylene trinitramine	RDX	2.11	43.9	0.587	21.9
Cyclotetramethylene tetranitramine	НМХ	1.65	28.9	0.666	33.3
1,3-dinitrobenzene	13DNB	0.519	40.1	0.496	24.8
2,4-dinitrotoluene	24DNT	0.612	40.2	0.424	21.2
2,6-dinitrotoluene	26DNT	1.15	52.4	0.524	26.2
N-Methyl-N-2,4,6- tetranitrobenzoaniline	TETRYL	0.556	44.5	0.731	20.2
Nitrobenzene	NB	10.7	54.9	2.41	27.4
Nitroglycerin	NG	NA	NA	4.00	200
Pentaerythritol tetranitrate	PETN	NA	NA	4.00	80
1,3,5-trinitrobenzene	135TNB	0.626	42.1	0.448	24.4
2,4,6-trinitrotoluene	246TNT	0.588	40.2	0.456	22.8

NA - Not applicable; ESE is not certified for these compounds in the aqueous matrix.

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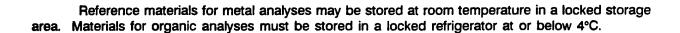
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TABLE 6-20 ESE CERTIFIED REPORTING LIMITS FOR CHEMICAL SURETY DEGRADATION PRODUCTS AND ASSOCIATED COMPOUNDS

Analyte	USAEC Acronym	Aqueous Limits	Reporting (µg/L)	Solid Repo (µg	
		Certified	Upper	Certified	Upper
1,4-Oxythiane	OXAT	1.98	39.5	0.856	17.1
Diisopropylmethylphosphonate	DIMP	10.5	210	0.114	4.57
Dimethylmethylphosphonate	DMMP	15.2	305	0.133	4.18
1,4-dithiane	DITH	1.11	22.2	0.571	11.4
Isopropylmethylphosphonic acid	IMPA	100	9000	2.1	40
Dimethyldisulfide	DMDS	1.14	22.8	0.692	13.8
p-Chlorophenylmethylsulfoxide	CMPSO	4.23	106	2.25	45.0
p-Chlorophenylmethylsulfone	CPMSO2	4.22	106	2.37	47.4
p-Chlorophenylmethylsulfide	CPMS	1.26	25.3	1.08	21.6
Methylphosphonic acid	MPA	128	9000	2.0	40
Thiodiglycol	TDGCL	187	4880	3.94	102
bis(2,4,6-trichlorophenyl)urea ^a	TCPU	NA	NA	0.8	20

ESE is not currently USAEC certified for TCPU; however, analytical quality control procedures will follow USAEC protocol.

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6.11 DATA VALIDATION, REDUCTION, AND REPORTING OF ANALYTICAL DATA

6.11.1 Collection

Data are initially collected, converted to standard reporting units, and recorded in standard formats by the project analysts. These project analysts conduct preliminary data analyses using a variety of methods and procedures. Because many analytical instruments are microprocessor controlled, some of the requisite analyses can be performed directly in the instrument's operating or outputting mode. Those instruments interfaced to stand-alone computers or microprocessors often permit data analysis programs to be written and modified to produce data formats specifically suited to end user requirements.

Data requiring manual recording, integration, and/or analysis may be converted to a more appropriate format prior to subsequent analyses. Through all stages and aspects of data processing, the data are double checked for translation or transcription errors and are initialed by both the recorder and the checker. The QA Manager or other designated individual not directly involved in the analysis reviews the data for acceptability.

6.11.2 Validation

Data validation is the process whereby data are determined to be of acceptable or unacceptable quality based on a set of predefined criteria. The criteria for the data is dependent upon the referenced sampling and analytical methodologies which includes the associated QA/QC requirements. The guidelines for the validation process used by USAEC is comparable to Laboratory Data Validation Functional Guidelines (Viar 1988b) and Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses (Viar 1988a). The National Functional Guidelines with Region III modifications (USEPA 1993a, 1993b) will be implemented for the validation of USEPA CLP data.

USAEC control charts will be maintained to provide a timely assessment of precision of measurement functions. USEPA CLP data packages will be constructed for the review of sample analysis. An independent review of all USEPA and USAEC data packages will be performed to ensure compliance with specified analytical, QA, and data reduction procedures; data reporting requirements; and required accuracy, precision, and completeness measures. The following items may be reviewed by QA Manager or USAEC, respectively, to validate the data:

- a. Sample holding times;
- b. Documentation that the analytical results are in control and within the certified (linear) range of the analysis;
- c. Documentation that data and calculations were checked by a reviewer who was not involved in the performance sampling, analysis, or data reduction;

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- d. Calibration data associated with specific methods and instruments:
- e. Routine instrument checks (calibration, control samples, etc.);
- f. Documentation on traceability of instrument standards, samples, and data;
- g. Documentation on analytical methodology and QC methodology;
- h. The potential presence of interferences in analytical methods (check of reference blanks and spike recoveries);
- i. Documentation of routine maintenance activity to ensure analytical reliability; and
- j. Documentation of sample preservation and transport.

All data generated will be assessed for accuracy, precision, and completeness. Data assessment techniques will include routine quality control checks and system audits.

Accuracy will be assessed from measurements of NIST SARMs or samples spiked with known concentrations of reference materials. The assessment for accuracy will be independent of the routine calibration process (e.g., reference materials will be obtained from independent sources and will be prepared independently).

6.11.3 Reduction

Data reduction frequently includes computation of analytical results from raw instrument data and summary statistics, including standard errors, confidence intervals, test of hypothesis relative to the parameters, and model validation.

Data reduction procedures that the laboratory will utilize address the reliability of computations and the overall accuracy of the data reduction. The numerical transformation algorithms used for data reduction will be verified against a known problem set to ensure that the reduction methods are correct.

The equations and the typical calculation sequence that should be followed to reduce the data to the acceptable format is instrument- and method-specific. Where standard methods are modified, data reduction techniques will be described in a report accompanying the data.

Auxiliary data produced for internal records and not reported as part of the analytical data include the following: laboratory worksheets, laboratory notebooks, sample tracking system forms, instrument logs, standard records, maintenance records, calibration records, and associated quality control. These sources will document data reduction and will be available for inspection during audits and to determine the validity of data.

Outliers will be identified by the USAEC control chart program, and the rationale used for data acceptance or rejection will be described and documented.

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6.11.4 Review

Validated data will be reviewed by the QA Manager for accuracy, precision, completeness, and comparability with respect to the field sampling activities. All field QC samples (i.e. rinse and trip blanks, field duplicates) will be reviewed and the environmental data will be evaluated based on the QC sample results. Any deficiencies will be investigated and reported in QA/QC section of the final report.

6.11.5 Reporting

Chemical data shall be reported in the USAEC Installation Restoration Data Management System (IRDMS). For chemical data obtained using USAEC methods, the analyst shall quantify each analyte in the method blank and spiked QC sample each day of analysis. Method blank data shall generally be reported as "less than" the CRL for each analyte. Values detected above CRL shall be reported as determined, with entry into the USAEC data management system in terms of concentration. Processing of additional sample lots will not occur until the results of the previous lots have been calculated, plotted on control charts as required, and the entire analytical method shown to be in control. For chemical data obtained using USEPA CLP methods, all environmental results and QA/QC sample results will be reported as Non USATHAMA Analytical Methods (NTAMs). All data qualifiers will be reported by ICF with results.

A detailed description of IRDMS is provided in Section 7.0 of this QAPjP.

SECTION 7.0

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7.0 INSTALLATION RESTORATION DATA MANAGEMENT SYSTEM

The Installation Restoration Data Management System (IRDMS) is an integrated system for the collection, validation, storage, retrieval, and presentation of USAEC Installation Restoration Program (IRP) and base closure data. IRDMS PCTool provides the ability to enter chemical, geotechnical, and map file data. Each contractor is supplied with the appropriate microcomputer-based software to allow for record entry, error checking, and quality control for chemical, geotechnical, and map file data. Records accepted by the local error checking program are then transmitted through a Bulletin Board System (BBS) AT&T Model 3B2 minicomputer, which is centrally located at USAEC's Edgewood, MD facility. Subsequent processing at the central site (duplicate error check) results in an elevation of the accepted records to a higher file "level" and the eventual updating of installation-specific data bases in a Pyramid system.

7.1 DATA MANAGEMENT

There are three levels of data recognized in the IRDMS. Level 1 consists of all files on the ICF KE microcomputer that have been entered or generated by the error checking program. The only Level 1 files that are present on the Pyramid system are program files. Program files are composed of several elements. An element may contain various contractor-written utilities or programs, add-streams, or other commonly used sets of commands.

It is anticipated that error-free files will be transmitted on a weekly basis to the Pyramid system. The ICF KE terminal will be linked to the network using software supplied by USAEC and a Hayes modem. Terminal usage logs will be established and maintained as a permanent record of communications. If communications cannot be established and maintained, ICF KE will seek optional means, where needed, for forwarding the data to USAEC. To verify acceptance, each file will be processed through an error checking program that is identical to the one on ICF KE's microcomputer. Accepted files will then be sent to the UNIVAC. Should any files fail this final error check, ICF KE will be notified and required to correct detected errors and retransmit the data.

Upon arrival at the Pyramid system, the files will be classified as Level 2 files. These records will be protected by write keys and, therefore, they may not be modified by ICF KE. They may be read by ICF KE, provided the appropriate read key is specified. All Level 2 files will be the responsibility of USAEC. Level 2 files will exist only until the data are loaded into the appropriate installation data base; normally within 10 working days.

Data in the installation data base are considered Level 3 data. They may be accessed by ICF KE using USAEC-supplied report programs and the appropriate read key; however, they are protected from changes by a write key. The installation data bases are the responsibility of USAEC.

Data management will begin when USAEC transmits a request for analytical services to the laboratory, stating the number, type, sample numbers, methods for analysis, and any other information necessary for the laboratory to plan a particular job. Data files of initial input information, including map location files, a certification status check, sample ID number, parameters, dates, etc. will be established

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as sample containers and chain of custody documentation are prepared for shipment to the field sampling team.

While in the process of collecting, documenting, packaging, and shipping samples to the laboratory, the field sampling team will transfer sample data from their notebooks to field parameter forms. Once the samples arrive at the laboratory, this information will be used to create Level 1 data files in the IRDMS. Status information (e.g., date sampled, date received, data extraction/analysis due) will form a part of the record.

Each step in the analytical process will result in updates to the data files. The operation performed (e.g., preparation, extraction, analysis, data review, data package prepared), the data obtained, and the date that each step was completed will be entered into the system and made available for status checks. The laboratory will validate the data, perform error-checking and correction using the USAEC routines, and transmit the Level 1 files to USAEC, via the 3COM communications network. Hard-copy documentation will also be transferred from the laboratory to USAEC.

Once the Level 1 files have been processed at USAEC, the PDC will transmit any required corrections, then generate a backup tape copy. This step will be completed within 50 days after the samples have been collected. The laboratory will archive copies of all analytical data, including original instrument magnetic tapes, in perpetuity. Records will also be maintained, so that historical summaries of all analyses may be generated by site, by client, or by sample type. Refer to Figure 7-1 for a summary diagram of how these data will be handled.

7.2 PROJECT DATA

Data for entry into the IRDMS and generated during this project will consist of geotechnical data and sampling/analytical data. The types, origin, IRDMS files, and handling of these data are described below.

7.2.1 Geotechnical Data

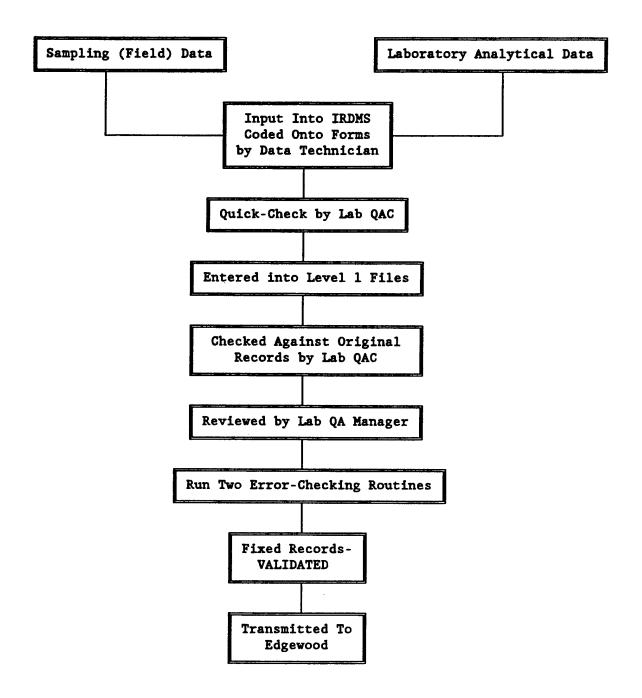
One IRDMS geotechnical data file will be generated by the Field Team activities during the TERA at APG. The Geotechnical Map File (GMA) will contain information about the location of all samples collected as part of the TERA.

7.2.2 Map File Data

The map file is a listing of sample sites and corresponding north and east coordinates. Map files must be created prior to entry of any other type of sample site data into the IRDMS. Before sampling is initiated, site coordinates are usually established and entered into the map file. A map file data form will be prepared from data contained in the field sampling logbooks. These data are entered into the computer by the Program Data Coordinator, and a computer printout of the file is checked and corrected by the Task Manager or designee. The data are submitted to USAEC in Level 1 and subsequently validated by the QA Supervisor. Once validated, this map file is elevated to Level 2. This must take place before any other data is processed.

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FIGURE 7-1
DATA MANAGEMENT SCHEME



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7.3 SAMPLING AND ANALYTICAL DATA

Data from analyses performed by the laboratory are input into various chemical data files, including CSW (surface water), CSE (sediment), CSO (soil data), and CQC (QC data). Data from sampling activities that are required by the laboratory will be submitted by the sampling team on field parameter forms along with the samples. The sampling organization will also be responsible for generation of all map files, as described above. A description of sampling and analytical data generation and manipulation is provided below.

Sampling data will be collected in the field in a permanently bound notebook (log). Portions of the information will be transferred to a three-part field parameter form. This information will include the site type, site ID, sampling date and time, field sample number, sample depth (if applicable), and the sampling technique. This form will accompany the samples to the laboratory so that the information can be encoded prior to sample analysis. A complete list of required information is presented in Table 7-1. In addition, each sample container will be annotated in waterproof ink with the installation name, sample number, sampling date, analytes, and preservatives. A chain-of-custody form will also be completed in the field and will accompany the samples to the laboratory, along with the field parameter form (see Appendix A for sample forms).

Collection of analytical data will begin when samples arrive at the laboratory. A laboratory technician will first verify that the samples noted on the chain-of-custody form coincide with the sample containers being delivered. If any containers are broken or missing, the chain-of-custody form will be annotated and the Field Operations Leader will be notified immediately. Samples will then be logged into a project-specific notebook and the computerized laboratory data management system according to parameter code, site ID, and laboratory sample number. The field parameter and chain-of-custody forms will then be submitted to a laboratory data technician for later correlation with the analytical results.

On receipt of the sample log information, the laboratory Quality Assurance Coordinator (QAC) will assign analytical lot numbers to the samples in accordance with USAEC procedures. The first three letters of the six-character sample code will designate the analytical lot, while the remaining three digits will indicate the sample number within the lot (e.g., AAB006 indicates the sixth sample in lot AAB). All quality control samples required for each analytical lot (e.g., method blank, control spike at two times the certified reporting limit (CRL), and two control spikes at ten times the CRL) will also receive USAEC sample numbers. The data technician will enter the sample information into the IRDMS to generate partially-completed data coding forms.

When the samples are taken from storage for analysis, the chain-of-custody (COC) form will be signed by the Data Analyst to acknowledge receipt of the samples for processing. When analyses are complete, the Data Analyst will reduce the data for QC samples to determine if the analyses were in control. The QC results will then be reviewed by the Laboratory Section Manager and forwarded to the QAC for verification. If the QAC agrees that the data are in control, the Data Analyst will be directed to proceed with data reduction for the samples. Concentrations of contaminants in extracts will be determined from instrumental responses of the extracts applied to the instrument calibration curve. The resultant concentration will then be modified by applying the appropriate dilution/concentration and sample weight or volume to obtain a final reportable concentration in the

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TABLE 7-1 LIST OF SAMPLING DATA

	Installation
	Field Sample Number
•	Matrix
•	Sampling Depth (if applicable)
•	Sampling Date and Time
	Sampling Location
	Method of Sampling
•	Analytes
•	Preservatives
•	Significant Observations
•	Printed Name and Signature of Sampler
•	Number of Samples Taken
	Temperature, pH, and Conductivity of Surface Water Sampled
	Number of Shipping Containers
	Date of Shipment

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original matrix. For soils, results will not be corrected for moisture; however, percent moisture is reported with the analytical results. Aqueous results will be reported in units of micrograms per liter and solid samples will be reported in micrograms per gram.

The data will contain no more than three significant digits and will be rounded to the appropriate number of significant digits, based on certification class and dilution, only after all calculations have been completed. When samples are diluted into a certified range, the reported concentration will contain one less significant digit than an undiluted sample. Values less than the certified reporting limit will be reported as "less than" the CRL. If a sample is diluted below the CRL, the value will be reported as "less than" the CRL multiplied by the dilution factor to more accurately reflect the observable limit. The dilution factor will be reported with the data. Method blank values will not normally be subtracted from sample results submitted to USAEC; however, method blank corrections may be made in accordance with the USATHAMA QA Program (1990).

When data reduction has been completed for the samples, all data (whether on magnetic media or hard-copy) will be transmitted to USAEC. The correlation of the analytical and field data will be performed by Potomac Research Incorporated (PRI). Table 7-2 lists the information that is required for the IRDMS. Further data processing is described in the next section.

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TABLE 7-2

INFORMATION REQUIRED FOR GEOTECHNICAL AND CHEMICAL DATA ENTRY INTO IRDMS

IRDMS DATA ENTRY ELEMENTS	GEOTECHNICAL DATA ENTRY	CHEMICAL DATA ENTRY
Installation	X	X
Laboratory		X
Sample		X
Test Method		X
Measurement Units		X
Analyst		X
Sample Number		X
File Name	X	X
Site Type	X	X
Site ID	X	X
Field Sampler Number	X	X
Sample Date	X	X
Sample Program		X
Sample Depth (cm)	X	
Sample Technique	X	
Lab Analysis Number		X
Sample Preparation Date		X
Analysis Date		X
Test Name		X
Measurement Boolean		X
Uncorrected Measurement Value		X
Dilution Factor		
Percent Moisture		X
Internal Standard Code		X
QC Test		X
QC Spike Value		X

SECTION 8.0

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8.0 SYSTEM CONTROLS

This section discusses document control, QC samples, control charts, and out-of-control conditions.

8.1 DOCUMENT CONTROL

The goal of the ICF KE Document Control Program is to ensure that all project documents issued or generated will be accounted for upon completion of the project. The program includes a numerical document control system, document inventory procedure, and a central filing system with a designated person(s) responsible for its maintenance.

All documents used or generated during the course of the project are accounted for and become a part of the project files upon completion of the task. These may include but are not limited to the following:

- a. Sample identification documents and field logbooks;
- b. Chain-of-custody record:
- c. Project Deliverables (i.e., Work Plans, audit reports, etc.);
- d. Analytical logbooks, laboratory data, calculations, graphs, strip charts, field logs, and software:
- e. Reports and correspondence material; and
- f. Photographs, maps, and drawings.

When an error is made on an accountable document, corrections are made by drawing a single line through the error and entering the correct information. The correction must also be initialed and dated. A brief explanation is provided explaining the reason the correction was made.

Controlled documents which are sensitive to timing or approvals will use a document control format in the upper right corner which includes:

- a. Document:
- b. Section number;
- c. Revision number;
- d. Date of revision; and
- e. Page__ of ___.

A distribution list of controlled documents will be maintained by the Program Data Coordinator (PDC), who will ensure that revisions are distributed to all addressees.

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After technical work on a task has been completed, all accountable documents generated or used for the task work will be assembled and placed in a secure storage location. All accountable task documentation will then be inventoried by the PDC.

8.2 LABORATORY QUALITY CONTROL SAMPLES

Laboratory QC samples are analyzed to provide quantitative evidence supporting the performance of the analytical system, and to demonstrate that the sensitivity is analogous to the level achieved during certification. The laboratory QC sample is prepared by the person conducting the first step of the analytical method. QC samples will be blind to the analyst conducting the actual analysis.

Laboratory quality control samples include the following:

- Method blank is a standard matrix sample to which no analyte of interest has been added that
 is processed in the same manner as samples, to ensure that the apparatus and reagents used
 are not contributing contaminants to the analysis.
- Replicate is a duplicate sample created in the laboratory that is extracted and analyzed in order to demonstrate the precision of the method of analysis.
- Surrogate standard is a pure compound added to every sample to monitor the recovery and to verify the efficiency of the extraction and analysis procedure. Recovery is the percent difference between the concentration spiked and the concentration quantitated by the method.
- Matrix spike is a known amount of target analyte added to the sample and which is then
 carried thorough the complete analytical method in order to demonstrate the accuracy of the
 method of analysis.
- Matrix spike duplicate is a duplicate of the matrix spike performed in order to demonstrate the precision of the spiking procedure.
- **Laboratory control standard** is a standard that can be traced to an alternate source than the working standard that is analyzed to verify the integrity of the working standard.
- **Homogenization blank** is a rinse blank performed on equipment used to homogenize soil and benthic tissue samples after decontamination procedures have been performed.

The number of laboratory QC samples analyzed is dependent upon the method of analysis. For specific information regarding the number of laboratory QC samples analyzed, please refer to the analytical method.

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8.3 CONTROL CHARTS

Where applicable, control charts will be used to monitor the trends and variations in the accuracy and precision of analytical analyses. The control chart shall contain the following:

- a. Title, analyte, method number, and laboratory name;
- b. Spike concentration;
- c. Three-letter lot designation and analysis date for each point along the abscissa;
- d. Percent recovery (X charts) or Range (R charts) along the ordinate;
- e. Upper and lower control limits; and
- f. Upper and lower warning limits.

Criteria and formats for control chart construction can be found in the USATHAMA QA Program (1990).

B.4 OUT-OF-CONTROL CONDITIONS

Situations arising from failure to adhere to standard operating procedures, policies, and protocols mandated by the USATHAMA QA Program (1990) have the potential to adversely affect data quality and affect investigation and or corrective action. All out of control situations for all project aspects will be investigated and appropriate corrective actions instituted. Areas in which operator error is normally associated with out-of-control conditions include:

- a. Failure to achieve calibration;
- b. Record keeping omissions;
- c. Improper sampling techniques;
- d. Improper sample storage and preservation; and
- e. Poor analytical protocols.

The detection of out-of-control conditions warrants some type of corrective action. Section 12.0 of this plan provides protocols for documenting corrective action.

SECTION 9.0

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9.0 PREVENTIVE MAINTENANCE

Instrument maintenance, both routine and preventive, will be performed as requested by USAEC. A preventive maintenance plan allows for periodic instrumentation checks for problems that occur frequently. The objective of a preventive maintenance plan is to rectify equipment problems before they become serious. Preventive maintenance also brings attention to those areas of the instrument susceptible to degradation from aging, toxic/corrosive attack, and clogging due to environmental factors.

Procedures for preventive maintenance are contained in each instrument's manual under the maintenance/troubleshooting sections. Each piece of equipment will have an associated standard operating procedure (SOP) detailing the calibration/maintenance instructions. Equipment failing calibration specification will be identified with a red warning label and will not be used for sample analysis.

Equipment requiring calibration will have an assigned record number which is permanently affixed to the instrument. A label will be affixed to each instrument containing the following information:

- a. Description;
- b. Manufacturer;
- c. Model number:
- d. Serial number:
- e. Date of last calibration or maintenance;
- f. Name of person who performed calibration or maintenance; and
- g. Date of next servicing.

9.1 CALIBRATION/MAINTENANCE FREQUENCY SCHEDULE

Schedules for calibration/maintenance must be accomplished at the manufacturer's recommended frequency, unless prior experience dictates a more frequent schedule. Should a schedule not be provided by the manufacturer, the calibration group servicing the equipment must provide a written calibration and maintenance frequency. A list of critical spare parts for field equipment is provided in the respective equipment operation's manual.

9.1.1 Field Equipment Calibration/Maintenance Frequency and Calibration Standards

For purposes of preventive maintenance, field equipment in storage will be calibrated by the ICF KE equipment manager according to the following schedule:

Photoionization Detectors:

HNu Pl-101: Every 30 days while in storage. HNu HW-101: Every 30 days while in storage. MICROTIP HL-200: Every 30 days while in storage.

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Conductivity, temperature, and pH meters (including Hydrolab®) are calibrated only in the field, refer to Section 5.6 for calibration of instrumentation in the field. The particular standards to which the PID and OVA are calibrated to by the ICF KE Equipment Manager are specified below:

Photoionization Detector Calibration Standards:

Benzene (C_6H_6) 1010 ppm +/- 1%, balance: Air. Benzene (C_6H_6) 100 ppm +/- 1%, balance: Air. Benzene (C_6H_6) 10 ppm +/- 1%, balance: Air. Isobutylene ($I-C_4H_8$) 100 ppm +/- 2%, balance: Air.

Analytical accuracy of all calibration gases is traceable to Standard Reference Materials (SRMs) from the National Bureau of Standards (NBS).

9.1.2 <u>Laboratory Calibration/Maintenance Frequency Schedule</u>

The contract laboratory will be responsible for maintaining calibration and maintenance of all laboratory equipment.

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10.0 RECORD KEEPING

Bound logbooks shall be utilized for all record keeping purposes both in the field and laboratory. It is assumed that the use of the bound book will result in a chronological sequence of data insertion. All logbooks will contain a unique document control number. If corporate controlled logbooks are used, the document control number will be on all pages. Non corporate controlled logbooks will be bound, and the document control number need only be contained on the document cover. All pages will be numbered, but numbered pages may be limited to pages with information.

To facilitate data validation, the person making an entry must sign and date the entry. All entries must be recorded in ink. Correction to entries shall be made by drawing a line through the incorrect entry, recording the correct information, and initialling and dating the corrected entry.

If computerized information is utilized, a hard copy which has been permanently affixed to the logbook will be acceptable as an original record of sampling and laboratory logging.

Logbooks containing information specific to the project shall be forwarded to USAEC at the end of the project. Should the need for corporate controlled logbooks arise, copies of all relevant logbook pages shall be submitted.

10.1 SAMPLING

Logbooks for sampling and field investigation purposes must meet the requirements specified by SOPs (Appendix A). They must be bound, and entries recorded in waterproof ink. The logbook must contain information to distinguish samples from each other. The following information should be included for each sample collected:

- a. USAEC project;
- b. Sequential field sample number;
- c. Matrix sampled;
- d. Sample depth;
- e. Sampling date and time;
- f. Specific sampling location:
- g. Method of sampling;
- h. Preservation techniques;
- i. Analytes of interest;
- j. Sampling observations;
- k. Results of field measurements;
- I. Printed name and signature of samplers;
- m. Date of shipment;
- n. Number of shipping containers; and
- o. Samples sent and carrier.

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10.2 LABORATORY RECORDS

10.2.1 Laboratory Logging

Once samples have been received by the laboratory, they shall be logged into a bound laboratory notebook. Information necessary for the logbook includes:

- a. Field sample number;
- b. Laboratory receipt date;
- c. Condition in which sample arrived;
- d. Analysis requested; and
- e. USAEC sample identification number.

10.2.2 <u>USAEC Sample Identification Numbers</u>

Data reporting to the USAEC Installation Restoration Data Management System (IRDMS) requires that each aliquot of a sample be assigned a six-character sample identification number. The number is comprised of two three-letter character designations. The first three characters define the analytical lot, which is based upon the number of samples capable of being processed in a 24-hour period. The last three characters pertain to the sequential order in which the instrumental analysis will be performed within the lot.

Different lot designations are used for each analytical method. Multi-analyte methods have the same lot designation for each analyte in a single sample aliquot. Should the contractor laboratory utilize an internal numbering system, the correlation to the USAEC sample identification number shall be provided in the logbook.

10.2.3 Analytical Records

10.2.3.1 Reference Materials. Bound logbooks must be maintained of all reference materials used for analytical purposes on the project. The record must include the following information:

- a. Date of receipt;
- b. Source;
- c. Purity;
- d. Composition;
- e. Storage conditions; and
- f. Expiration date.

10.2.3.2 Sample Handling. All personnel involved in performing any aspect of the analytical protocol must maintain a record of the activities in a bound logbook. Although this logbook must be specific to the operation, it need not be operator specific. The logbook should be signed and dated daily and contain the following information:

- a. Samples handled;
- b. Standards used;

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- c. QC samples prepared;
- d. Procedures used; and
- e. Resultant calculations.

10.2.3.3 Instrument Operation. Each instrument must have a dedicated logbook. Information in the logbook must reflect routine and emergency maintenance activities, tuning, absolute and chemical curve calibration, and all analytical activities conducted on the instrument. A new page must be started daily during equipment operation. Information to be included for each page consists of:

- a. Date, operator, and project name;
- b. Description of any instrument maintenance or modification;
- c. Tuning and calibration activities;
- d. Instrument settings;
- e. Instrument operating conditions; and
- f. Samples analyzed.

The use of automated data acquisition systems will require recording a reference to the data file for each standard or sample.

Hard copy data output from integrators and chromatograms should have the following information clearly evident on the printout:

- a. Analysis date and time;
- b. Test name and sample number;
- c. Reference to the calibration curve used for quantitation;
- d. Logbook reference to recorded analytical activities; and
- e. Identification of chromatographic peaks.

SECTION 11.0

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11.0 AUDITS

This section discusses performance and system audits used to monitor the capability and performance of the total measurement system to evaluate the quality of operation in the field and in the laboratory. A performance audit is a planned independent check of the operation of a system to obtain a quantitative measure of the quality of data generated, and involves the use of standard reference samples or materials which are certified as to their chemical composition or physical characteristics. System audits are of a qualitative nature and consist of on-site review of a system's quality assurance system and physical facilities for sampling/analysis, calibration, and measurement. ICF KE will be responsible for auditing the contract laboratory and for auditing field activities.

11.1 FIELD SYSTEM AUDITS

A field QA audit will be conducted during the first few days of field activities to determine if the field teams are following protocols delineated in this QAPjP. The audit will be performed by ICF KE's QA Manager or an appropriate designee. The field QA audit will monitor to determine whether requirements stated in the QAPjP are being met. The QA Manager will check for performance of the following items, at a minimum, during the course of the audit:

- Copies of the Health and Safety Plan, workplan (ICF KE 1992), and QAPjP are on-site and accessible to the sampling teams;
- The field instruments are of the proper type, and have been properly calibrated. All calibrations have been recorded in a permanent bound logbook.
- All information listed in the SOP for field logbooks is recorded in a permanent bound log in indelible ink;
- Samples are collected from the least contaminated to the most contaminated locations;
- Sample collection procedures are performed as per the QAPjP using the proper sampling equipment, sample containers, and preservatives. Samples are placed on ice immediately after collection;
- Sample bottles are properly packaged as per the QAPjP for shipment (including sealing with appropriate custody seals);
- Chain-of-custody forms include all information listed in the SOP, and;
- Sampling equipment is properly decontaminated between sample locations, as detailed in the QAPjP.

During the audit, actions will be taken on the spot by the QA manager to ensure that field sampling is conducted in accordance with the QAPjP and workplan (ICF KE 1992). The QA manager will document any deficiencies encountered during the audit and any actions taken in the field to

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correct potential problems. Results of the audit will be maintained at the ICF KE office in Abingdon, Maryland as part of the QA documentation.

11.2 LABORATORY SYSTEM AUDITS

The contract laboratory will be evaluated at a frequency dictated by the laboratory's performance, and will include a quality assurance on-site evaluation to inspect the Contractor's facilities to verify the adequacy and maintenance of instrumentation, the continuity of personnel meeting experience or education requirements, and the acceptable performance of analytical and QC procedures. The items to be monitored include, but are not limited to, the following:

- a. Size and appearance of the facility;
- b. Quantity, age, availability, scheduled maintenance and performance of instrumentation;
- c. Availability, appropriateness, and utilization of SOPs;
- d. Staff qualifications, experience, and personnel training programs;
- e. Reagents, standards, and sample storage facilities;
- f. Standard preparation logbooks and raw data;
- g. Bench sheets and analytical logbook maintenance and review; and
- h. Review of the laboratory's sample analysis/data package inspection procedures.

A formal audit report will be provided to the Project Manager, Analytical Task Manager, and USAEC. Results of the audit will be documented and maintained as part of the QA documentation.

11.3 PERFORMANCE AUDIT

EPA Region III may choose to submit a spiked performance evaluation (PE) sample to the sampling team leader. This sample will be submitted to the laboratory with the other environmental samples collected at APG. The sample will be analyzed for those analytes requested by EPA. The results of the analysis will be used by USEPA to determine laboratory accuracy.

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12.0 CORRECTIVE ACTION

Corrective action will be initiated through the development and implementation of routine internal quality control checks. Specific limits beyond which corrective action is required will be established for each system. Corrective action requirements will be implemented in response to deficiencies encountered during system audits or failure to adhere to the QAPjP.

To enhance the timeliness of corrective action and thereby reduce the generation of unacceptable measurement data, problems identified by assessment procedures will be resolved at the lowest possible management level. Problems that cannot be resolved at this level will be reported to the QA Manager for resolution. The QA Manager will determine at which management level the problem can best be resolved, and will notify the appropriate manager. Weekly progress reports will detail all problems and subsequent resolutions.

Steps comprising a closed-loop corrective action system include:

- a. Defining the problem;
- b. Assigning responsibility for problem investigation;
- c. Investigating and determining the cause of the problem:
- d. Assigning responsibility for problem resolution; and
- e. Verifying that the resolution has corrected the problem.

Documentation on the corrective action requirements, the assignment of responsibility for corrective action, due dates for completion of corrective action, and validation of completion will be maintained. Such documentation will be reviewed during system audits. Figure 12-1 is a proposed report form for use by all project staff to document the resolution of all corrective actions.

12.1 LINE OF COMMUNICATION FOR CORRECTIVE ACTION

If a visitor to the site, including USEPA and State of Maryland oversight personnel, observes a health and safety or quality assurance problem at the site, or a deviation from the work plan, then the visitor should express their concern to the ICF KE Field Operations Leader or Task Manager. The ICF KE personnel will either agree with the visitor, correct the perceived problem or deviation, and continue working, or will disagree with the visitor and continue working. The visitor's comments will be documented in the appropriate field logbook. If the visitor's comment is not acted upon by the ICF KE Field Operations Leader or Task Manager, then the visitor may communicate with the facility environmental coordinator, who may decide to contact the USAEC Project Officer. It should be noted that the ICF KE Field Operations Leader and Task Manager will comply with directions given by the USAEC Project Officer but not necessarily with visitors to the site or regulatory oversight personnel.

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FIGURE 12-1

CORRECTIVE ACTION REPORT FORM

CORRECTIVE ACTION REPORT FORM Date of Problem: _____ Originator:____ Description of Problem and Effect on System: ______ Title _____ Date: Persons Notified: Description of Gorrective Action:____ Person Completing Action: Signature______Title_____Date____ Approval:_____ Title____ Date__

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13.0 QUALITY CONTROL REPORTS

The following documents and deliverables will be submitted to DSHE in support of the project work performed at APG.

- a. Pre-certification and certification data packages;
- b. Audit reports:
- c. Weekly QA/QC reports during field activities;
- d. IRDMS submissions;
- e. Monthly status reports of QC activities:
- f. QC charts (during periods of analytical analyses);
- g. Logbooks;
- h. QA section of the project final report; and
- i. Project final report

USAEC will be responsible for the final storage and security of all data files at a location on APG.

If changes are to be made to the QAPjP prior to the close of the project, the proposed changes will be submitted to the Project Officer at the Directorate of Safety, Health, and Environment (DSHE), John Paul.

SECTION 14.0

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